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Laboratory Procedures

analysis for wastewater treatment plant operators

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LABORATORY PROCEDURES

ANALYSIS FOR WASTEWATER TREATMENT PLANT OPERATORS

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LABORATORY PROCEDURES

ANALYSIS FOR WASTEWATER TREATMENT PLANT OPERATORS

by: David Vietti

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

Construction Grants
Operation and Maintenance
911 Walnut Street
Kansas City, Missouri 64106
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INTRODUCTION

The need for basic straight-forward procedures and qualified wastewater laboratory analysts is increasing at a rapid rate. With more and more emphasis being placed upon the quality of treated waste discharged into the interstate and intrastate streams, lakes, rivers and waterways, the need for better laboratory control is apparent.

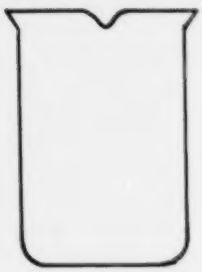
This wastewater laboratory manual is furnished by the Environmental Protection Agency as an aid to the laboratory analyst for making wastewater analyses. It is not meant to be the ultimate answer for the most precise and accurate tests. However, the procedures contained herein for the most widely used parameters in a treatment plant are of the highest precision.

The most accurate and precise results are obtained by following the procedures found in the latest edition of Standard Methods and WQO Methods For Chemical Analysis of Water and Wastes. Many of the tests found in these texts are not economically feasible for many wastewater treatment plants, and this manual is meant to provide alternate test procedures which will provide results of sufficient accuracy.

In addition to this laboratory manual, the analyst should have available the latest editions of Standard Methods for the Examination of Water and Wastewater, ASTM Standards, and WQO Methods for Chemical Analysis of Water and Wastes, and the latest edition of Laboratory Manual for Chemical and Bacteriological Analysis of Water and Sewage by Theroux, Eldridge and Mallman.

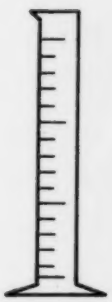
GLASSWARE

ERLENMEYER FLASK



BEAKER

BURETTE



GRADUATED CYLINDER

B.O.D. BOTTLE

PIPETTE

CLEANLINESS, STERILIZATION, CHEMICALS, CONTAMINATION, EQUIPMENT, AND TECHNIQUE

Glassware is an essential part of most chemical analysis procedures. It can be cleaned by rinsing with an acid cleaning mixture made up by adding 1 liter of concentrated sulfuric acid, slowly with stirring, to 35 mls saturated sodium dichromate solution. Acetone is a very good organic solvent and can be used as a cleaning solution also. After cleaning the glassware, rinse thoroughly with warm tap water and finally distilled water.

A clean container is usually defined by the layman as one which is visibly free from dirt or foreign material. Since many cleaning solutions are not bactericides, a visibly clean piece of equipment does not necessarily mean that the equipment is free of microorganisms since they are not visible to the naked eye. The cleaning agent used might leave a film on the equipment which could cause erroneous results. Sterilization of equipment as practiced in the microbiological laboratory is accomplished by heating or steaming the object to be sterilized sufficiently long to kill all living organisms. This is usually done with the use of an autoclave. The bottles used to send in water samples need to be sterilized so that foreign matter and living microorganisms will not be present.

The reagents used in the determinations of chemical quality of water and sewage have been compounded to give accurate results and have been standardized for the purpose intended. Contamination of reagents by using dirty glassware or by using the same pipette for several reagents will cause erroneous results. The purpose of laboratory control thus is defeated. Rinsing of all glassware in distilled water prior to use is necessary. All droplets of water should be shaken from the apparatus to prevent continuous dilution of reagents. Reagents can be purchased in standardized form from chemical houses. This is slightly more expensive than is the preparation of reagents in the laboratory.

Work in the chemistry laboratory consists of combining chemicals under controlled conditions to establish a knowledge of comparison between a standard and an unknown. Certain procedures have been devised whereby simple tests will provide the knowledge desired. The laboratory as known to water and sewage works operators should be considered a tool to assist in control of plant processes.

Many chemicals, when combined, react violently. Knowledge in the field of chemistry is necessary to predict what combinations of chemicals or compounds will react as desired. A few precautionary measures should be followed in the laboratory:

1. Follow instructions
2. Combine only those materials as instructed
3. Consider the laboratory as a tool and use as directed
4. Keep equipment clean
5. Record findings immediately
6. Do not contaminate reagents

Much of the technique employed in a chemistry laboratory can be learned from observing and practicing the various operations and procedures. Many of the chemicals used are strong acids and bases which are harmful to eyes, skin and clothing. The first and most important technique to practice is caution. The following items should be remembered and practiced in all laboratory work:

1. When adding an acid to an aqueous solution permit the acid to enter by sliding down the side of the container slowly. Never add water to acid.
2. Do not apply suction with the mouth when filling a pipette with a strong reagent. Rather dip the tip of the pipette into the reagent and cover upper opening with the finger before removing.
3. If glassware slips, let it fall. An attempt to catch falling glassware might result in a dangerous cut.
4. If in doubt of the contents do not sniff strongly at the mouth of a container.

5. To neutralize strong acid on skin or clothing wash with tap water and apply dilute Ammonium Hydroxide solution.

6. To neutralize strong alkali or base chemical on skin or clothing wash with excess of tap water and apply dilute acetic or hydrochloric acid.

7. If any chemical gets in the eye wash with excess of tap water and see your physician immediately.

The analytical balance is a precision instrument that plays a very important role in a laboratory. With the aid of the balance, solutions of the proper strength may be prepared to be used in the accurate determination of a particular substance. Determinations may be made directly by employing weighing procedures, and it is the standard for accuracy in the laboratory.

The balance is an expensive apparatus that must be used with care because of its relative delicateness. Rough use will damage the balance and decreases its accuracy or even impair its operation. The balance should be centrally located in an even-temperatured room and carefully guarded against radiations from heating apparatus and excessive vibrations. When not in use the beam should be supported by the beam rests and the pans should be supported by the pan rests.

SAMPLING

PURPOSE

The analysis cannot be any better than the sample itself. Therefore, the sample must be as correct as possible.

Samples are taken to allow the operator to test for the amount of improvement in water quality by each unit in the plant and for the plant as a whole. In order to further evaluate the efficiency of his plant, he must know the condition of the raw sewage entering his plant, the effluent from the plant and the condition of the receiving stream above and below the effluent discharge.

Normal sampling points are listed below. These may be changed or modified to suit the individual plant.

1. Influent (Raw Sewage) - At a convenient point prior to any treatment where one can obtain a representative sample.

2. Effluent of primary tanks - This point should be selected near the lower end of the effluent channel to allow thorough mixing of effluent from entire unit.

3 Aeration tank.

4. Effluent of trickling filters.

5. Effluent of final clarifier.

6. Receiving stream - At least fifty yards upstream from entry of effluent.

7. Receiving stream - At least fifty yards below entry of effluent.

The points may be established to fit the individual plant, but should be so arranged to give a uniform and true picture of the operation of each unit of the plant and the influence of the plant on the condition of the stream.

METHODS

For most of the tests, the samples should be collected in a wide-mouth bottle of a predetermined capacity. Each grab sample for the composite should be at least 300 mls in size. A holding device should be constructed so that the collection bottle can be held below the surface, mouth pointed in the direction of flow.

RATIO OF SAMPLING TO FLOW

In order to accurately evaluate the data, it is desirable that the volume of sample collected each time be related by simple ratio to the total flow at that particular moment.

COLLECTION

The importance of collecting and handling sewage samples in the most careful manner cannot be overemphasized. The procedures and equipment used in the laboratory are assumed to be the most accurate and precise obtainable. If the results from the tests are to be accurate, precise and representative, the same precision and accuracy must be exercised in the collection, handling and storage of the samples prior to the actual laboratory procedures. It is even more important that samples be collected during the time that any unit of the plant is out of operation. The results of the plant operation during a breakdown are often valuable evidence in the event of a lawsuit.

FREQUENCY

The more frequent the sampling, the more complete the results. If it is impossible to run samples quite frequently, a five-day schedule has the advantage of permitting titrations for the DO of present samples and BOD of the previously incubated samples, all at one set-up.

If the sampling is to be done on a weekly basis, the samples should be taken on different days of the week in order that any daily variations in the sewage characteristics may be found.

COMPOSITES

A grab sample taken at any particular hour of day or night does not give a true picture of the over-all plant operation. Therefore, if the grab samples are collected and composited hourly or at some suitable time interval over the working day or the 24-hour period in volumes related by ratio to the total flow, we have then a Composite Sample, which closely represents the conditions which existed over the period in which samples were collected.

$$\frac{\text{Rate of flow at time of collection} \times \text{total sample needed}}{\text{Number of portions} \times \text{average rate of flow}} = \text{amount of single portion}$$

NOTE: A composite sample is composed of two or more portions added together. It may be collected over any desired period of time and the portions may be collected at any desired intervals. To obtain a representative sample each single portion must be measured proportional to the rate of flow at the time of collection. The rate of flow may be in either gpm or gpd.

EXAMPLE: Samples are collected at intervals of four hours over a 24-hour period making a total of six portions. The rate of flow at the successive sampling times are 1.5 mgd - 1.2 mgd, 2.0 mgd - 1.3 mgd - 1.6 mgd and 1.4 mgd. The average rate of flow is 1.5 mgd; the required total amount of sample is 1200 mls.

Find the size of each portion in mls

1. $\frac{1.5 \times 1200}{6 \times 1.5}$ = 200 mls size of first portion

2. $\frac{1.2 \times 1200}{6 \times 1.5}$ = 160 mls size of second portion

3. $\frac{2.0 \times 1200}{6 \times 1.5}$ = 267 mls size of third portion

4. $\frac{1.3 \times 1200}{6 \times 1.5}$ = 173 mls size of fourth portion

5. $\frac{1.6 \times 1200}{6 \times 1.5}$ = 213 mls size of fifth portion

6. $\frac{1.4 \times 1200}{6 \times 1.5}$ = 187 mls size of sixth portion

$\frac{}{1200 \text{ mls}}$ = total sample

STORING

The samples should be stored at 4°C. This retards any further bacterial action until you are ready to run the sample. The volume of each hourly or bi-hourly sample should be calculated to fill a gallon container about 3/4 full.

FULL BOTTLE WINKLER METHOD FOR DISSOLVED OXYGEN TEST

PURPOSE

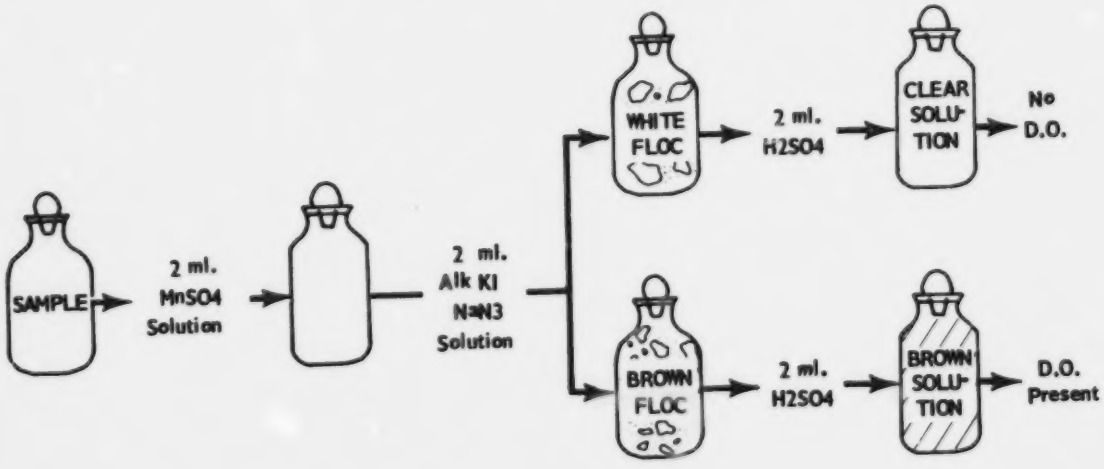
The test measures the oxygen present as a gas in solution. This information may be valuable in:

1. Studies of septicity of sewage
2. Odor control
3. Operation of pre-aeration units
4. Operation of secondary treatment facilities
5. Pollution control.

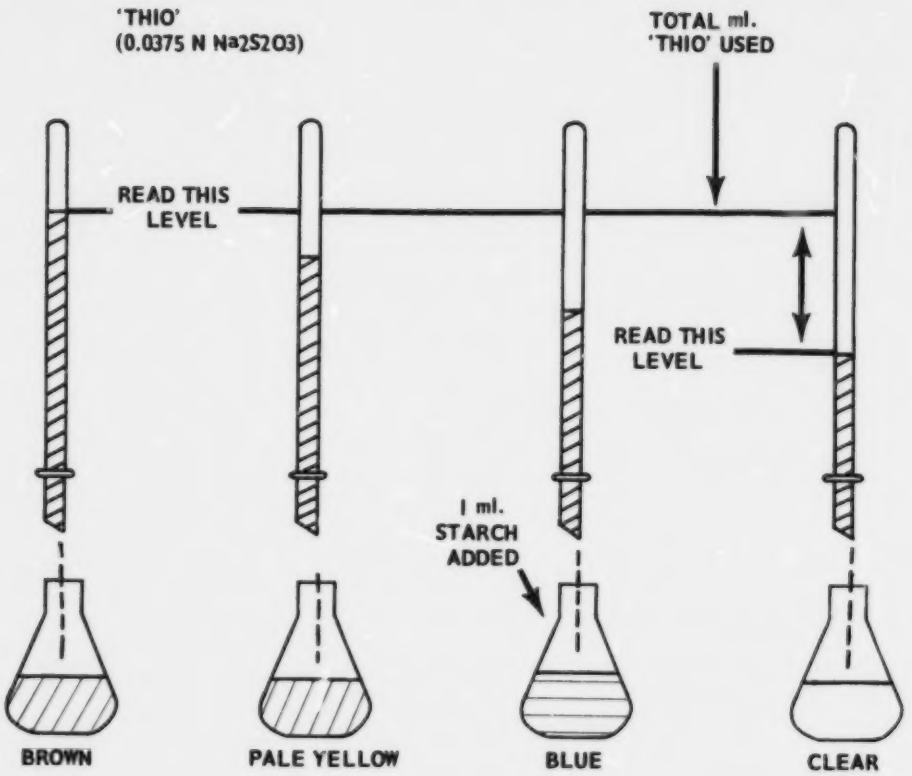
The azide full bottle modification of the Winkler Method is the most accurate and precise dissolved oxygen (DO) test. The azide eliminates the nitrites sometimes found in sewage. The nitrites will cause an error in the DO value if the azide is not used.

OUTLINE OF PROCEDURE

1. Addition of test materials (reagents) to sample, see diagram.



2. Titration:



3. Recording of results. Record volume of 0.0375 N sodium thiosulfate (thio) used in the titration.
(1 ml thio = 1 mg/l or ppm of Dissolved Oxygen)

PREPARATION OF TEST MATERIALS (REAGENTS)

CHEMICALS REQUIRED (All chemicals should be of "analytical reagent grade.")

1. Manganous sulfate, $MnSO_4.4H_2O$ or $MnSO_4.2H_2O$, or $MnSO_4.H_2O$
2. Sodium hydroxide or potassium hydroxide, NaOH or KOH
3. Sodium iodide or potassium iodide, NaI or KI

4. Sodium azide, NaN_3
5. Sulfuric acid, H_2SO_4, concentrated
6. Soluble starch
7. Sulfamic acid, NH_2SO_2OH, technical grade
8. Copper sulfate, $CuSO_4 \cdot 5H_2O$
9. Sodium thiosulfate, $Na_2S_2O_3 \cdot 5H_2O$
10. Chloroform, $CHCl_3$
11. Potassium dichromate, $K_2Cr_2O_7$
12. Acetic acid, concentrated
13. Distilled water.

PREPARATION

Manganous Sulfate Solution ($MnSO_4$ solution)

1. Dissolve 480 grams $MnSO4.4H_2O$ or 400 grams $MnSO_4.2H_2O$ or 364 grams $MnSO_4.H_2O$ in distilled water. This is difficult to dissolve. Use electric stirrer if possible.
2. Add enough distilled water to make one liter and mix thoroughly.

Alkaline Iodide-Sodium Azide Solution (KI NaN_3)

1. Dissolve 500 grams NaOH or 700 grams KOH and 135 grams NaI or 150 grams KI in distilled water. Each substance should be dissolved separately and in small amounts of distilled water. Mix them when they are cool. CAUTION: Add water slowly with stirring, avoid breathing fumes, and avoid bodily contact with the solution. Heat is produced when the water is added and the solution is very caustic.
2. Dissolve 10 grams NaN_3 in 75 ml distilled water. CAUTION: NaN_3 is poisonous.

3. Add the NaN_3 solution with constant stirring to the cooled solution of alkaline iodide.

4. After the cooled solutions are mixed, add enough distilled water to make a final volume of 1 liter and mix thoroughly.

Sulfuric Acid, concentrated (H_2SO_4)

Handle carefully, since this material will burn hands and clothes. Rinse affected parts with tap water to prevent injury.

Starch Solution

1. Take 5 to 6 grams of Arrowroot or soluble starch and add the least quantity of cool distilled water necessary to make a paste.

2. Pour this emulsion into 1 liter of boiling water.

3. Allow to boil a few minutes and settle overnight.

4. Use clear supernatant.

5. Add 10 mls chloroform and keep refrigerated.

6. Stable for about 1 month.

Sodium Thiosulfate Stock Solution ($Na_2S_2O_3$) - (0.75 N)

1. Dissolve 372.30 grams of $Na_2S_2O_3 \cdot 5H_2O$ in 1500 mls of boiled and cooled distilled water.

2. Add distilled water to make 2 liters and mix thoroughly.

3. Add 10 mls chloroform.

Working Sodium Thiosulfate Standard Solution - (0.0375 N)

1. Take 50 mls of sodium thiosulfate stock solution and add enough distilled water to make 1 liter. Mix thoroughly.

2. Add 10 mls chloroform.

3. Stable for about 1 month.

Copper Sulfate - Sulfamic Acid Inhibitor Solution

1. Dissolve 32 g sulfamic acid in 475 mls distilled water.
2. Dissolve 50 g copper sulfate in 500 mls water.
3. Mix the two solutions together and add 25 mls concentrated acetic acid. Bring up to 1 liter and mix thoroughly.

Potassium Dichromate Stock Solution ($K_2Cr_2O_7$) - (0.375N)

1. Dissolve 18.39 grams of $K_2Cr_2O_7$ in distilled water and add enough distilled water to make exactly 1 liter. Mix thoroughly.

Working Potassium Dichromate Solution - (0.0375N)

1. Take 100 mls of Potassium Dichromate Stock Solution and add enough distilled water to make 1 liter. Mix thoroughly.

Standardization

1. Add 250 mls of distilled water to a 500 ml, wide mouth, E-flask.
2. Dissolve approximately 2 grams KI in the distilled water.
3. Add 2 mls of concentrated H_2SO_4.
4. Pipette exactly 10.00 mls of 0.0375N $K_2Cr_2O_7$ into the solution.
5. Allow to stand in the dark for 10 minutes. The brown color which is developed is due to the liberation of iodine in solution.
6. Titrate this iodine with the sodium thiosulfate being standardized. When the color is pale yellow, add 1 or 2 mls of starch solution and continue adding thiosulfate solution until the blue color disappears.
7. Record burette readings.

8. Assuming the burette reading was 9.50 ml of sodium thiosulfate used, the following calculations are done to find the amount of distilled water which must be added to a volume of sodium thiosulfate working solution to make it 0.0375N. For the following example, it is assumed that 800 ml of sodium thiosulfate working solution is being adjusted to 0.0375N.

$$[(ml)\ K_2Cr_2O_7]\ [(N)\ K_2Cr_2O_7] = [(ml)\ Na_2S_2O_3]\ [(N)\ Na_2S_2O_3]$$

From step 4 10.0 ml of 0.0375N $K_2Cr_2O_7$ were used.

$$[10\ ml]\ [0.0375N] = [9.50\ ml]\ [(N)\ Na_2S_2O_3]$$

$$[(N)\ Na_2S_2O_3] = 0.03947$$

9. $$[800.00\ ml]\ [0.03947] = [(ml)\ Na_2S_2O_3]\ [0.0375N]$$

$$[842.03] = [(ml)\ Na_2S_2O_3]$$

842.03 ml - 800 ml = 42.03 ml

42.03 ml of distilled water are to be added to the 800.00 ml of sodium thiosulfate working solution to give the desired normality of 0.0375N.

HOW TO MAKE THE TEST

1. Fill completely a 300 ml BOD bottle with the sample to be analyzed without allowing air to get into the bottle.

2. By holding the tip of the pipette below the surface of the liquid add:

   (1) 2 mls manganous sulfate solution,

   (2) 2 mls alkaline-iodide-azide solution.

3. Replace stopper, avoiding trapping air bubbles, and shake well. Repeat shaking after floc has settled halfway. Allow floc to settle again, about three-quarters of the way down from the top.

4. Remove stopper and add 2 mls of concentrated sulfuric acid down the neck of the bottle. Be sure to hold pipette above the surface of the liquid.

5. Mix to dissolve the floc. Handle carefully to prevent acid burns.

6. If solution has no yellowish brown color, or is only slightly colored, add 1 or 2 mls of starch solution. If no blue color develops, there is zero DO. If a blue color develops proceed

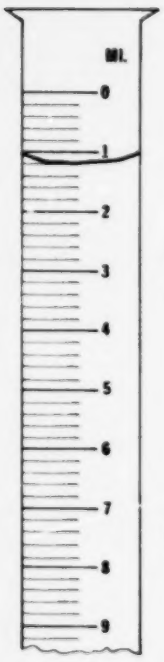
as in Step 7.

7. If solution is yellowish brown, pour into a wide mouth, 500 ml, Erlenmeyer flask, and titrate with 0.0375N sodium thiosulfate.

8. Add the thio until the color becomes pale yellow, then add 1 or 2 mls of starch solution and continue adding thio until the blue color disappears.

9. Record the number of mls of thio used.

10. Reading the burette:



MENISCUS 1.2 ml. IS THE READING

UPPER SECTION ONLY OF BURETTE

When the burette is to be used, the initial reading must be taken. The correct place to look is the bottom of the curve that the surface of the liquid forms. This curve is called the meniscus. After seeing where the liquid level is, record the result and proceed to add the thiosulfate solution (titrate). When the required amount of solution has been added, that is, when the blue color disappears, the final burette reading is made and recorded. Read the bottom of the meniscus as before. Subtract the initial reading from the final reading. This difference represents the net volume in milliliters (ml.) of solution used.

11. Calculation of the DO:

If the brown solution (Step 7) is titrated with the 0.0375N thio, then:

DO in mg/liter (or ppm) = ml of thio used.

12. Discussion

The sample for the dissolved oxygen test is usually collected in the bottle that will be used in the test. Extreme caution must be used to avoid contact of the sample with the air. The sample must be prepared immediately after collection.

DISSOLVED OXYGEN KITS AND PROBES

Portable kits and probes are available for field work. These kits are satisfactory for operation control analysis. They may be obtained from companies that supply laboratory equipment.

INHIBITOR FLOCCULATION MODIFICATION DISSOLVED OXYGEN TEST

Copper Sulfate - Sulfamic Acid Flocculation Modification Dissolved Oxygen Test

PURPOSE

This modification is used for biologic flocs, such as activated-sludge mixtures, which have high oxygen utilization rates.

How To Make The Test

1. Add 10 ml copper-sulfamic acid inhibitor to a 1 quart wide-mouth bottle.
2. Add the sample to the bottle, at least 500 mls, stopper, and mix by inversion.
3. Allow the suspended solids to settle quiescently and siphon the relatively clear supernatant liquor into DO bottle.
4. Continue the sample treatment as rapidly as possible by the Full Bottle Winkler Method For Dissolved Oxygen Test on Page 13.

SOLUBILITY OF OXYGEN IN FRESH WATER TABLE

Temperature		Dissolved Oxygen p.p.m.
°C	°F	
0	32.0	14.6
1	33.8	14.2
2	35.6	13.8
3	37.4	13.5
4	39.2	13.1
5	41.0	12.8
6	42.8	12.5
7	44.6	12.2
8	46.4	11.9
9	48.2	11.6
10	50.0	11.3
11	51.8	11.1
12	53.6	10.8
13	55.4	10.6
14	57.2	10.4
15	59.0	10.2
16	60.8	10.0
17	62.6	9.7
18	64.4	9.5
19	66.2	9.4
20	68.0	9.2
21	69.8	9.0
22	71.6	8.8
23	73.4	8.7
24	75.2	8.5
25	77.0	8.4
26	78.8	8.2
27	80.6	8.1
28	82.4	7.9
29	84.2	7.8
30	86.0	7.6

BIOCHEMICAL OXYGEN DEMAND (BOD) TEST

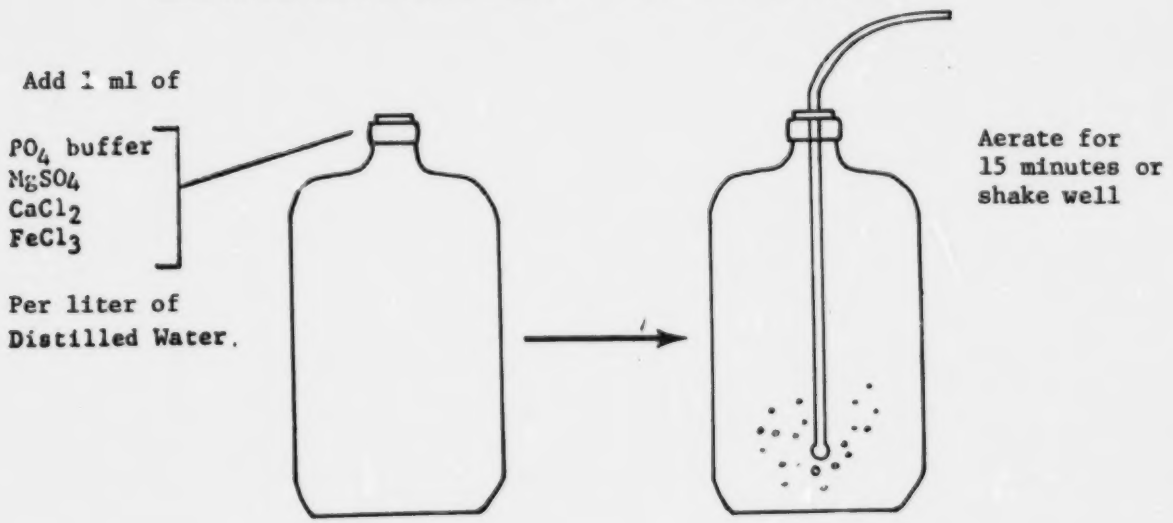
PURPOSE

The test measures primarily the organic polluting material in a sample. This information may be valuable in:

1. Studying the organic (pollutional) load on a plant or receiving stream
2. Determining the efficiency of sewage treatment
3. Pollution control

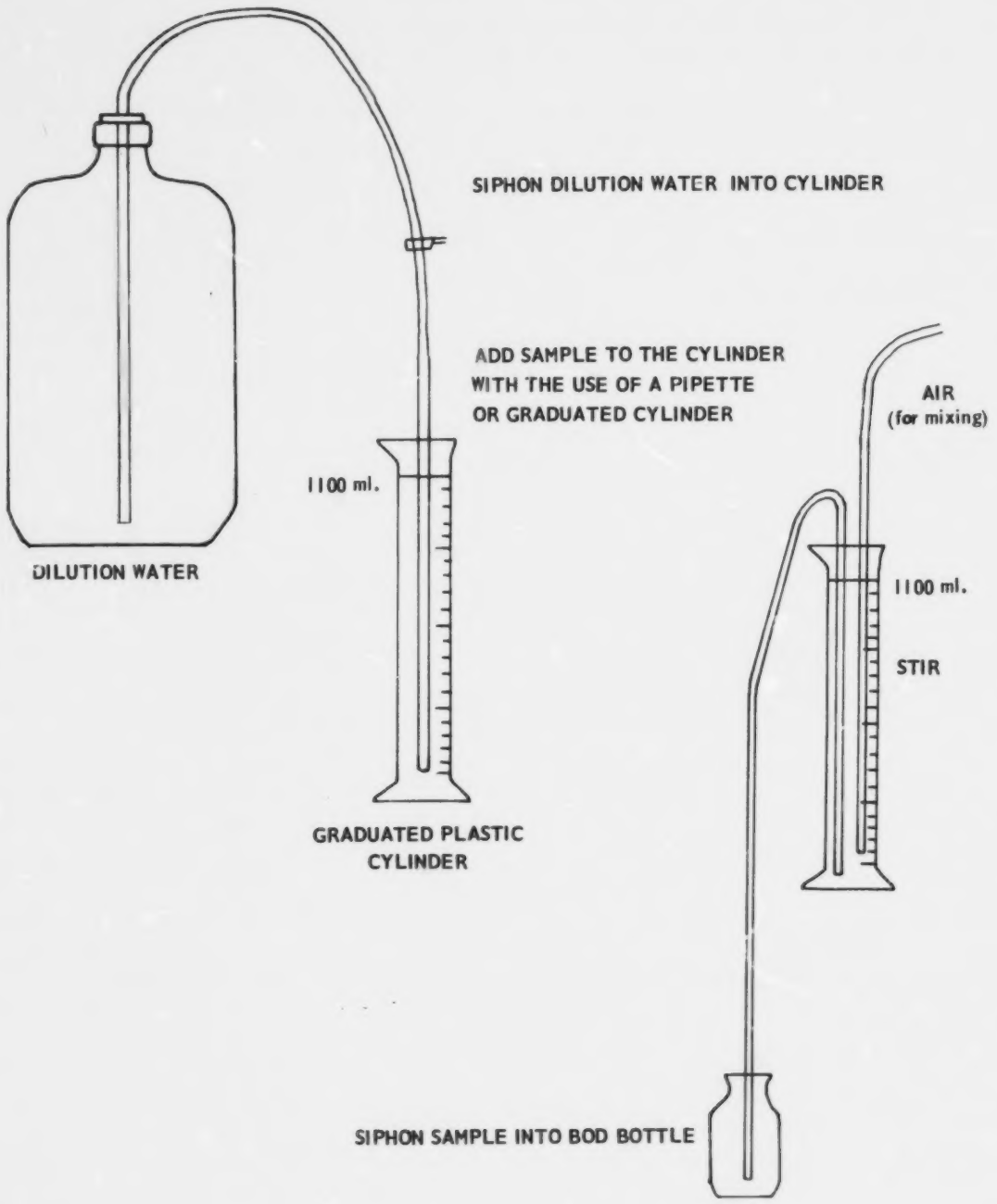
OUTLINE OF PROCEDURE

1. Preparation of dilution water:
   Aerate distilled water for 15 minutes or shake well



2. Pretreatment of samples, if indicated conditions exist.

3. DILUTION PROCEDURE



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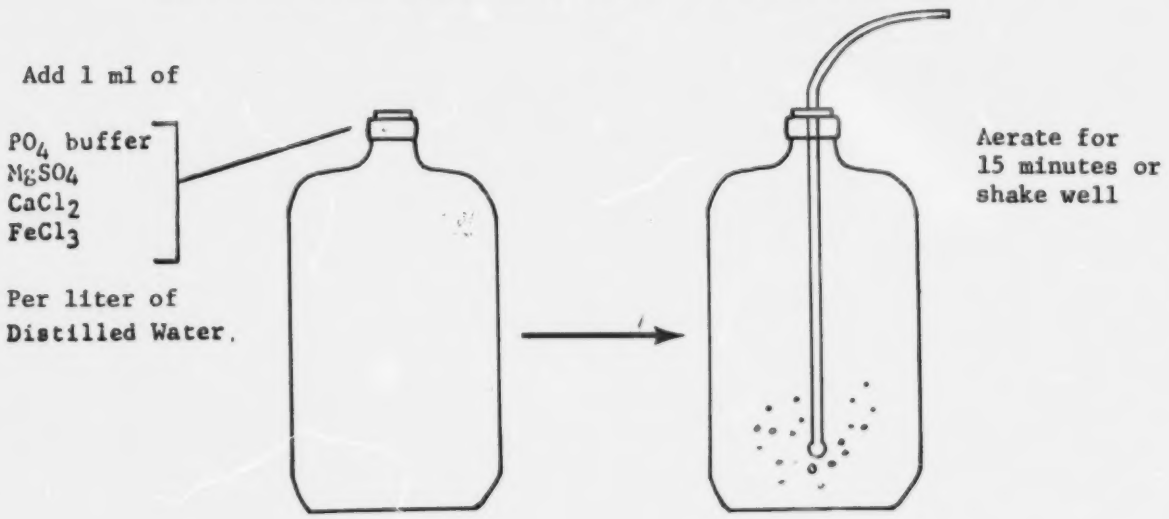
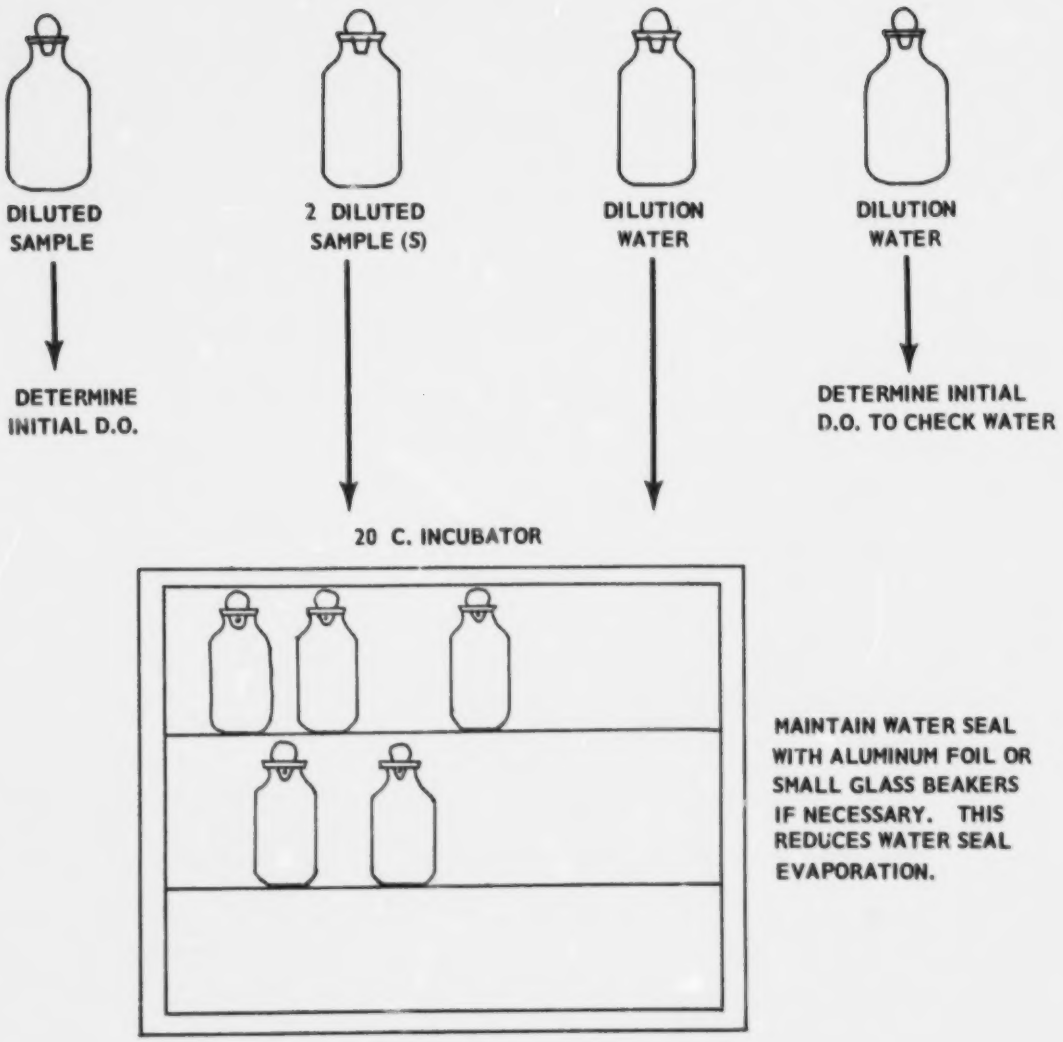


2. Pretreatment of samples, if indicated conditions exist.

4. IMMEDIATE D.O. DETERMINATION AND INCUBATION


DILUTED SAMPLE
2 DILUTED SAMPLE (S)
DILUTION WATER
DILUTION WATER
DETERMINE INITIAL D.O.
DETERMINE INITIAL D.O. TO CHECK WATER
20 C. INCUBATOR
MAINTAIN WATER SEAL WITH ALUMINUM FOIL OR SMALL GLASS BEAKERS IF NECESSARY. THIS REDUCES WATER SEAL EVAPORATION.

DETERMINE D.O. AFTER 5 DAYS INCUBATION

PREPARATION OF REAGENTS

CHEMICALS REQUIRED (All chemicals should be of "analytical reagent grade")

1. All chemicals and reagents for making dissolved oxygen determination
2. Potassium acid phosphate, KH_2PO_4
3. Potassium dibasic phosphate, K_2HPO_4
4. Sodium dibasic phosphate, $Na_2HPO_4 \cdot 7H_2O$
5. Ammonium chloride, NH_4Cl
6. Magnesium sulfate, $MgSO_4 \cdot 7H_2O$
7. Calcium chloride, $CaCl_2$ (anhydrous)
8. Ferric chloride, $FeCl_3 \cdot 6H_2O$

PREPARATION

Phosphate Buffer Solution

1. Dissolve 8.5 grams KH_2PO_4, 21.75 grams K_2HPO_4, 33.4 grams $Na_2HPO_4 \cdot 7H_2O$, and 1.7 grams NH_4Cl in about 500 mls distilled water.
2. Add distilled water to make 1 liter and mix thoroughly.
3. Stable for about 1 month.

Magnesium Sulfate Solution

1. Dissolve 22.5 grams $MgSO_4 \cdot 7H_2O$ in distilled water.
2. Add distilled water to make 1 liter and mix thoroughly.

Calcium Chloride Solution

1. Dissolve 27.5 grams anhydrous $CaCl_2$ in distilled water.
2. Add distilled water to make 1 liter and mix thoroughly.

Ferric Chloride Solution

1. Dissolve 0.25 grams $FeCl_3 \cdot 6H_2O$ in distilled water.

2. Add distilled water to make 1 liter and mix thoroughly.

How To Make The Test

1. Make sure that all the equipment and glassware are thoroughly clean.

2. Preparation of Dilution Water:

   (1) Store distilled water at 20°C for at least 24 hours. This can be accomplished in an incubator set at 20°C.

   (2) Bubble air through the volume of distilled water needed for the samples for approximately 15 minutes.

   (3) To the volume of distilled water add 1 ml of each of the following reagents per liter of distilled water:

      a. Phosphate buffer

      b. Magnesium sulfate

      c. Calcium chloride

      d. Ferric chloride.

   (4) Aerate the dilution water for approximately 15 minutes.

3. Pretreatment of Sample:

   (1) The sample must not contain residual chlorine. If the residual is high, take a 100 ml portion of the sample, add about 2 grams KI and 1 ml of concentrated H_2SO_4. Titrate with 0.0375N Sodium thiosulfate, using starch as an indicator just as in the DO test. Add to the sample itself the amount of 0.0375N Sodium thiosulfate just determined as necessary to neutralize the residual chlorine per 100 ml of chlorinated sewage sample. Mix well and after 10 minutes check a portion to be sure all residual chlorine is gone. Use this for the BOD determination.

(2) The sample must not be supersaturated with oxygen. If the sample has a dissolved oxygen of more than 9.2 mg/liter (parts per million, ppm) at 20°C, it is supersaturated. Shaking a bottle partially filled with the sample, or bubbling air through it for several minutes will remove the excess oxygen and the sample may then be used without further treatment.

4. Dilution Procedure:

Strong sewage must be diluted to give accurate results in the BOD test. Accurate results can be obtained by making the dilutions as follows in the ranges noted:

Sewage Strength BOD_5	Recommended Dilution %	Dilution Factor	ml Sewage in 1100 ml
From 1-7	No Dilution	1	1100 ml
From 2-14	50%	2	550 ml
From 4-28	25%	4	275 ml
From 5-35	20%	5	220 ml
From 10-70	10%	10	110 ml
From 20-140	5%	20	55 ml
From 50-350	2%	50	22 ml
From 100-700	1%	100	11 ml
From 200-1400	1/2%	200	5.50ml
From 400-2800	1/4%	400	2.75ml

After the approximate strengths are estimated:

Raw is usually between 200 and 400 mg/l BOD_5
Primary effluent between 30 and 230 mg/l BOD_5
Plant effluent between 10 and 50 mg/l BOD_5

The ml sewage can be determined from the end column above. Place the needed amount of sewage in a one liter graduated cylinder marked off at 1100 mls. Then fill the cylinder with BOD dilution water to the 1100 ml mark. Mix by bubbling air through. During this mixing the air bubbles have only a minimum effect. From this point on, however, one must exert every precaution to prevent any air bubbles in bottles.

From the graduated cylinder fill (3), 300 ml BOD bottles by siphoning from the cylinder and placing the tubing in

the bottom of the BOD bottles filling to the point of overflowing. Before inserting the stoppers be certain no small air bubbles exist on sides of the bottles. The best way to prevent any air bubbles is to have the bottles clean and free of all grease. Insert glass stopper and twist to seal. Maintain water seal during incubation period.

Record the bottle numbers on the record sheet along with the per cent dilution made. Place the two to be incubated into the incubator. The third sample is ready to be titrated to determine the amount of Dissolved Oxygen in the sample.

BOD results are most accurate when the oxygen in the sample is just half consumed. If less than 20% or over 70% of oxygen is consumed the results are of questionable accuracy. If this happens when the next dilutions are made, the dilutions should be increased or decreased to try to obtain results in which approximately half of the oxygen is consumed. A good "rule of thumb" is at least 2 mls of depletion and 1 ml of oxygen left.

EXAMPLE FOR BOD CALCULATION

A 1/2% dilution has been made for a sewage with a strength of between 200 and 1400 mg/l. 7.6 milliliters of 0.0375 normal sodium thiosulfate were used in the initial titration on the dilution. 4.0 milliliters of the sodium thiosulfate were used in the incubated dilution.

What is the BOD?

Initial	7.6	
-Incubated	4.0	
	3.6	
Multiply by	200	
	720	mg/l BOD_5

DISCUSSION

1. The biochemical oxygen demand determination is a measure of the amount of oxygen required to oxidize the organic matter in a sample in 5 days at 20° centigrade.

2. The collection of the BOD sample must follow a standard procedure. The same sampling points are used for each successive sample.

3. A composite sample will be most representative of the sewage to be tested. Eight hour composites, hourly, should be the minimum.

4. The test consists of the determination of dissolved oxygen prior to and following a period of incubation.

5. If the oxygen demand of the sample is greater than the available dissolved oxygen then a dilution must be made.

6. The amount of dilution depends upon the oxygen demand. A series of dilutions are required for unknown sewage samples.

7. Good overall operation of a secondary plant will usually remove 85 to 95 per cent of the BOD.

RELATIVE STABILITY — METHYLENE BLUE TEST

PURPOSE

The test for relative stability determines qualitatively the stability of sewage or a treated effluent, namely the percentage of the organic solids in the sewage which had been decomposed or digested by the action of biological organisms and converted into inert or stable chemical compounds not subject to further decomposition.

If all the available oxygen is consumed in a short period of time, the sewage under examination contains a large amount of undigested organic matter and therefore has a low stability value.

If the blue color remains for twenty or more days, complete digestion or stability of all the sewage matter in the sample can be assumed.

PREPARATION

Methylene Blue Solution

1. Dissolve 0.5 grams $C_{16}H_{18}ClN_3S\text{-}3H_2O$ in about 500 mls distilled water.

2. Add distilled water to make 1 liter and mix thoroughly.

HOW TO MAKE THE TEST

1. Clean a 300 ml DO bottle and rinse thoroughly.

2. Immerse the bottle in the liquid to be sampled and completely fill with as little agitation as possible. Chlorinated effluents cannot be used.

3. Add 0.8 ml of the methylene blue solution below the surface of the liquid and mix by inversion.

4. Restopper the bottle so that no air bubbles remain under the stopper.

5. Place in an incubator maintained at 20°C.

6. Observe daily and record the number of days or fractions of days that elapse before the blue color disappears.

Relative-Stability Numbers

Time required for decolorization at 20°C, days	Relative stability, per cent	Time required for decolorization at 20°C, days	Relative stability, per cent
0.5	11	8.0	84
1.0	21	9.0	87
1.5	30	10.0	90
2.0	37	11.0	92
2.5	44	12.0	94
3.0	50	13.0	95
4.0	60	14.0	96
5.0	68	16.0	97
6.0	75	18.0	98
7.0	80	20.0	99

SETTLEABLE, TOTAL AND SUSPENDED SOLIDS DISCUSSION

DISCUSSION

1. The settleable solids test is a measure of the amount of solids in ml per liter which will settle in a given period of time in an Imhoff cone or a graduated cylinder.

2. The sample should be a composite sample, although timed grab samples are often used with the Imhoff cone. The settleable solids of the mixed liquor using a graduated cylinder is always a grab sample.

3. The test using an Imhoff cone gives the results of settling in ml per liter and can be used to calculate the efficiency of settling tanks, as well as to calculate the amount of sludge which needs to be removed from settling tanks.

4. The settleable solids test using a graduated cylinder is used to determine the settleability of the mixed liquor in an activated sludge plant and in the calculation of the sludge index.

5. Total solids in sewage include suspended, dissolved, settleable, and organic as well as inorganic solids. The test is made in the following manner: An evaporating dish is weighed and placed on a steam or water bath. 50 or 100 ml of the sample is placed in the dish and evaporated. The sample and dish is then dried in the oven, cooled in the disiccator and weighed. The increase in weight x 1,000,000 divided by the ml of sample is equal to the mg/l of total solids.

6. The results of suspended solids tests may be used to evaluate the efficiency of the plant or the units in a plant. These are the solids in suspension that may be removed by filtering. A typical domestic sewage of 1000 ppm of total solid will contain about 300 mg/l suspended solids and approximately 85% of these solids, or more, will be organic solids.

7. Suspended solids may be determined by the Gooch Crucible method or by the use of a spectrophotometer. Suspended solids by the spectrophotometer method can be made in approximately 5 to 10 minutes time, where as the Gooch Crucible method will take about 2 or 3 hours or more. The Gooch Crucible method, however, is the more reliable and preferred test. The procedure for the spectrophotometer method is as follows:

   a. Shake sample well and pour about 700 ml into the cylinder of a blender.

   b. Blend for 90 seconds and transfer blended liquor to a battery jar and stir.

   c. While stirring siphon about 25 ml into the cuvette.

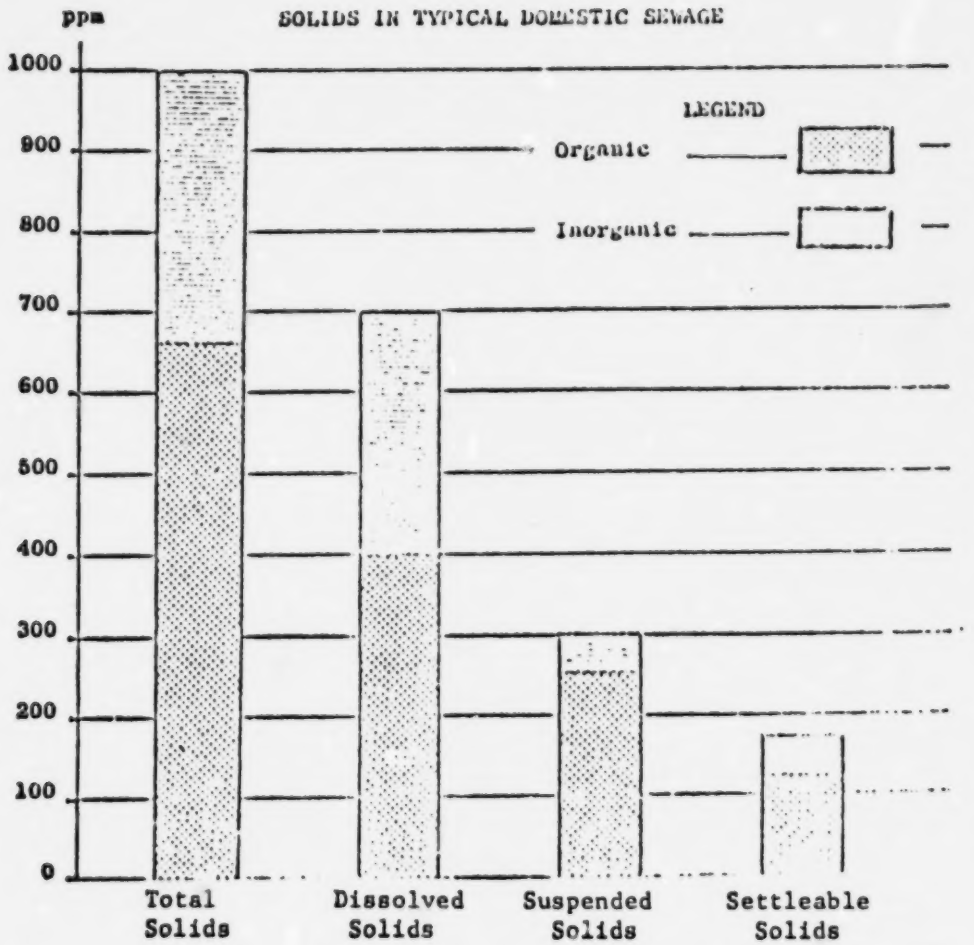
   d. With a 10,000 A setting on the spectrophotometer, read the transmittance or absorbance scale using distilled water as a blank.

   e. Just before reading, invert the cuvette gently to make sure all of the particles are in suspension, (Do not shake!).

   f. Read concentration of suspension solids from prepared graph.

   g. References: Determining Suspended Solids Using a Spectrophotometer, Sewage and Industrial Wastes, October, 1959.

   h. Dissolved solids are important because about 70% of total solids are in a dissolved state and cannot be removed with primary treatment. The greater portion of the organic load on secondary treatment units is dissolved solids.


ppm
SOLIDS IN TYPICAL DOMESTIC SEWAGE
1000
900
800
700
600
500
400
300
200
100
0
LEGEND
Organic
Inorganic
Total Solids
Dissolved Solids
Suspended Solids
Settleable Solids

SETTLEABLE SOLIDS TEST

Imhoff Cone

Settleable Solids: The determination of settleable solids in sewage may be accomplished by use of an Imhoff cone. The Imhoff cone (as illustrated) is made of glass or pyrex, is cone shaped and holds one liter when filled to the graduation mark near the top. The apex of the cone is graduated in milliliters, usually 0 to 40 ml.

To determine the efficiency of a settling tank or other plant unit two Imhoff cones are necessary, as samples to both the influent and effluent must be tested. The samples used in this test may be either grab, timed grab, or composite.

PROCEDURE

1. Fill Imhoff cone to mark with well mixed sample of sewage influent.
2. Fill another Imhoff cone to mark with well mixed sample of sewage effluent.
3. Allow to stand for a period corresponding to detention time in settling tank. (Usually two hours).
4. After sample has been standing for about three-fourths of total time, dislodge material clinging to sides of cone by giving it several twists, being careful not to disturb solids already settled to bottom.
5. At the end of total time, read results and record as milliliters per liter.

CALCULATION OF RESULTS (Example)

Milliliters per liter in influent	9.0
Less	
Milliliters per liter in effluent	.5
Milliliters per liter removed	8.5

To find amount of settleable solids removed in percentage, the following formula may be used:

$$\frac{\text{Milliliters per liter removed} \times 100}{\text{Milliliters per liter in influent}} = \text{percent removed}$$

$$\frac{8.5 \times 100}{9.0} = 94.4\%$$

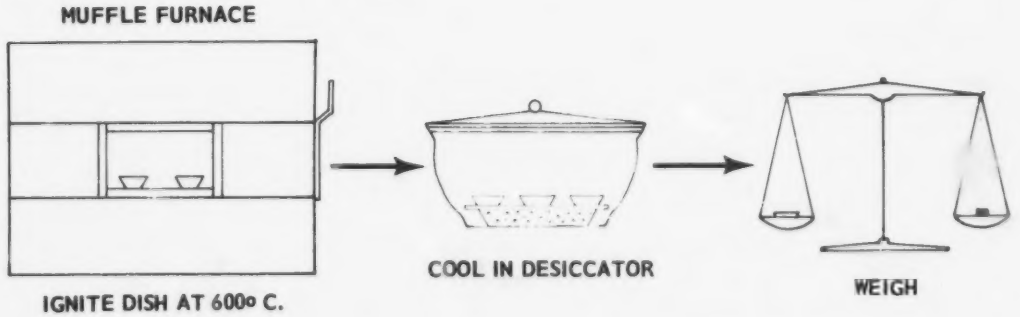
TOTAL SOLIDS TEST

PURPOSE

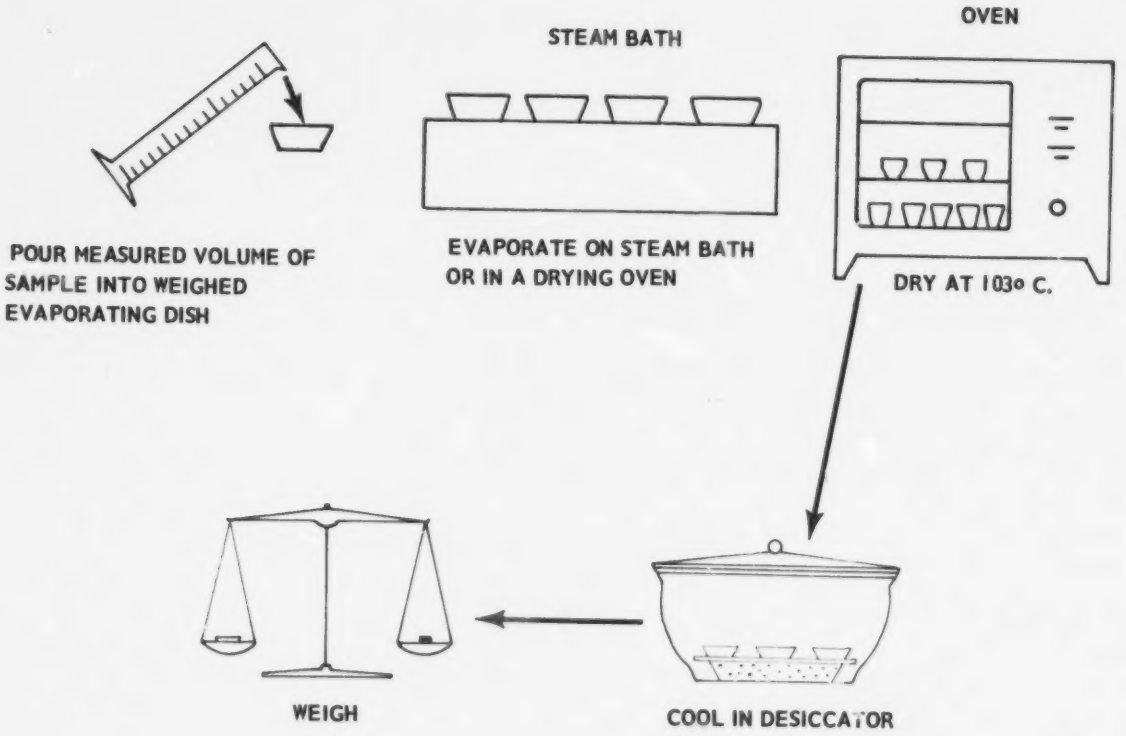
1. The test measures the amount of suspended and dissolved materials.
2. It may be used in studying plant loading and efficiency.

OUTLINE OF PROCEDURE

1. PREPARATION OF EVAPORATING DISH



2. TREATMENT OF SAMPLE


STEAM BATH
OVEN
POUR MEASURED VOLUME OF SAMPLE INTO WEIGHED EVAPORATING DISH
EVAPORATE ON STEAM BATH OR IN A DRYING OVEN
DRY AT 103° C.
WEIGH
COOL IN DESICCATOR

3. Recording of results

   (1) record weight of evaporating dish

   (2) record weight of dish with sample

   (3) record volume of sample

HOW TO MAKE THE TEST

1. Ignite evaporating dish at 600°C. (If volatile solids are not to be determined, the dish may be dried instead of ignited).

2. Cool in desiccator for 20 - 30 minutes.

3. Weigh.

4. Measure a 100 ml portion of well mixed sample in a graduated cylinder.

5. Pour sample into ignited evaporating dish.

6. Evaporate to dryness on a steam bath or in a drying oven. If a steam bath is used, the dish must be given a final drying in a drying oven at 103°C.

7. Cool dish in desiccator for 20 - 30 minutes.

8. Weigh and record weight.

9. Calculation of total solids:

   mg/liter (or ppm) total solids

   = (weight in mg of evaporating dish with sample - weight in mg of dish) $\times \frac{1000}{\text{ml of sample}}$

VOLATILE SOLIDS TEST

PURPOSE

1. The test measures the amount of volatile solids, that is, the solids which are largely organic in nature and can be destroyed by burning.

2. It may be used in studying

   (1) plant loading

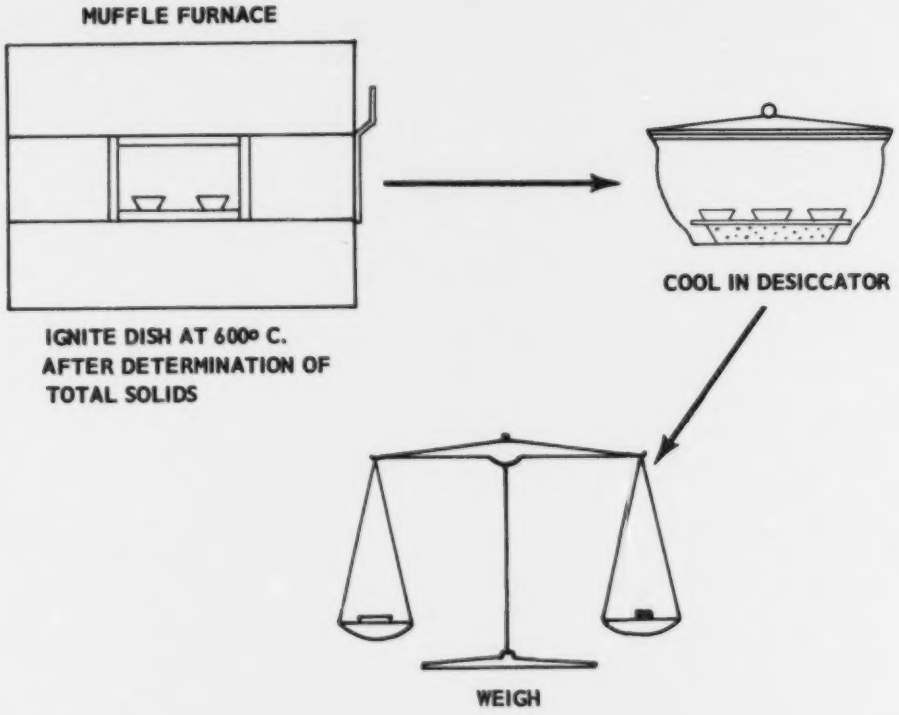
   (2) digestor loading

   (3) active material needed for biological treatment by activated sludge

Volatile solids may be determined on either total or suspended solids.

OUTLINE OF PROCEDURE

I. VOLATILE TOTAL SOLIDS



TOTAL VOLATILE SOLIDS SLUDGE TEST (SHORT CUT)

1. Weigh a prepared evaporating dish on the balances. Record weight.
2. Place a 100 gram weight on the balance.
3. Pour 100 grams well mixed sludge sample into the evaporating dish.
4. Record the weight of the dish plus the 100 grams.
5. Evaporate to dryness, ignite at 600°C.

6. Cool in desiccator.

7. Place on balances, add the weight of the dish. Add weights to determine weight of sludge.

8. The weight of the dry sludge in grams is equal to the percent total solids.

Example: Assume the evaporating dish weighs 75 grams. The dish and sample would weigh 175 grams. After evaporation the dish and sample weigh 85 grams.

The percent total solids = weight of dish and sample - weight of dish = 85 - 75 = 10 grams = 10 percent total solids.

Ignite and cool percent ash =

$$\frac{\text{(Weight of evaporating dish and sample - Weight of dish) } 100}{\text{Weight of evaporated sample}}$$

Assume the weight of sample and dish is 75 grams after evaporation.

$$\text{Percent Ash} = \frac{(75 - 70)\ 100}{10} \qquad \text{Percent ash} = 50$$

CENTRIFUGE METHOD FOR SUSPENDED SOLIDS TEST

PURPOSE

In this method the suspended solids are determined by centrifuging tubes containing mixed liquor at a specific rate of spinning for a definite period of time, reading the volumes of the sludge directly in per cent from the graduations on the centrifuge tubes, and multiplying this reading by a factor to convert to mg/l (ppm).

Outline of Procedure

Apparatus Required:

1. Centrifuge, clinical, with 4-place head for 15-ml tubes.

2. Centrifuge tubes, API, 12.5 ml capacity graduated in per cent.

3. Six beakers, low form, 250 ml capacity.

How To Make The Test

1. Thoroughly mix the sample of the activated sludge sample and pour about 100 mls into a 250 ml beaker.

2. Immediately pour the mixed liquor into two centrifuge tubes up to the 100 per cent mark.

3. Centrifuge the tubes at about 2500 rpm for exactly 15 minutes.

4. When the centrifuge comes to a stop, read the volumes of the sludge in the tubes directly in per cent from the graduation on the outside of the tubes.

5. Multiply this reading by a factor to roughly convert to ppm (mg/l). The factor will range from 600 for a "young" large floc sludge to 1000 for an "old" small floc sludge.

Example

Reading on the centrifuge tubes 4.5%
Multiplying reading by 800 gives. 3600ppm

Notes

More accurate results may be obtained from data showing direct relationships between actual results of suspended solids by the Gooch Crucible Method and the centrifuge tube readings.

GOOCH CRUCIBLE METHOD FOR SUSPENDED SOLIDS TEST

PURPOSE

This method is applicable to surface water, domestic and industrial wastes, and saline waters. The practical range of the determination is 20 mg/l to 20,000 mg/l. A well-mixed sample is filtered through a standard glass fiber filter, and the residue retained on the filter is dried to constant weight at 103-105°C. Non-homogenous particulates such as leaves, sticks, fish and lumps of fecal matter should be excluded from the sample. Too much residue on the filter will entrap water and may require prolonged drying.

Outline of Procedure

Apparatus Required:

1. Glass fiber filter discs, 4.7 cm or 2.2 cm, without organic binder, Reeve Angel type 984H, Gelman type A, or equivalent.

2. Filter holder, membrane filter funnel or Gooch crucible adapter.

3. Suction flask, 500 ml.

4. Gooch crucibles, 25 ml (if 2.2 filter is used).

5. Drying oven, 103-105°C.

6. Desiccator.

7. Desiccant.

8. Analytical balance, 200 g. capacity, capable of weighing to 0.1 mg.

How To Make The Test:

1. Insert the disc into the bottom of a suitable Gooch crucible.

2. While vacuum is applied, wash the disc with three successive 20 ml volumes of distilled water. Remove all traces of water by continuing to apply vacuum after water has passed through.

3. Dry Gooch crucible and filter in an oven at 103-105°C for one hour. Remove to desiccator and store until needed. Weigh immediately before use.

4. Assemble the filtering apparatus and begin suction. Shake the sample vigorously and rapidly transfer 100 mls to the funnel by means of a 100 ml volumetric cylinder. If suspended matter is low, a large volume may be filtered.

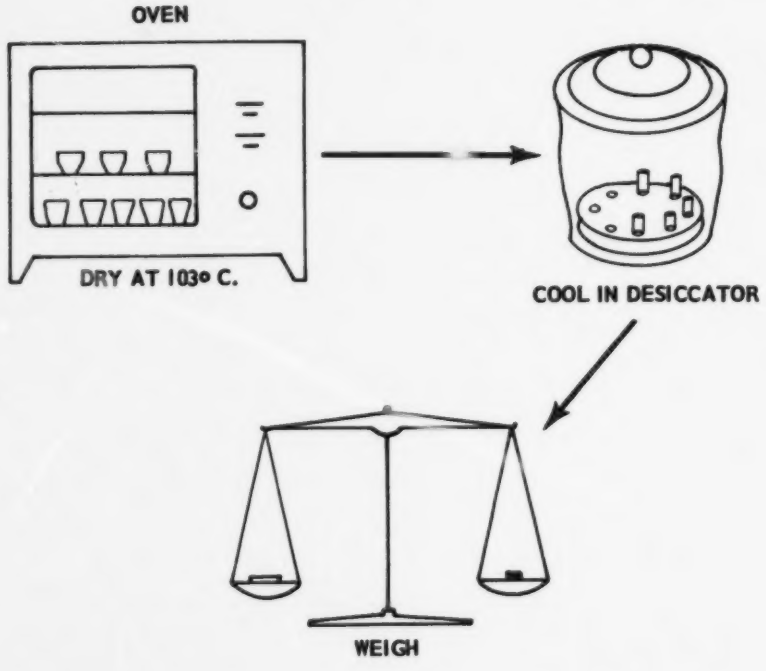
5. Place in drying oven and dry at 103-105°C to constant weight (usually overnight).

6. Calculations:

$$\text{Suspended Solids (mg/l)} = \frac{(\text{wt. of filter + residue} - \text{wt. of Filter}) \times 1000}{\text{ml of sample filtered}}$$

Outline of Procedure (Graphically)

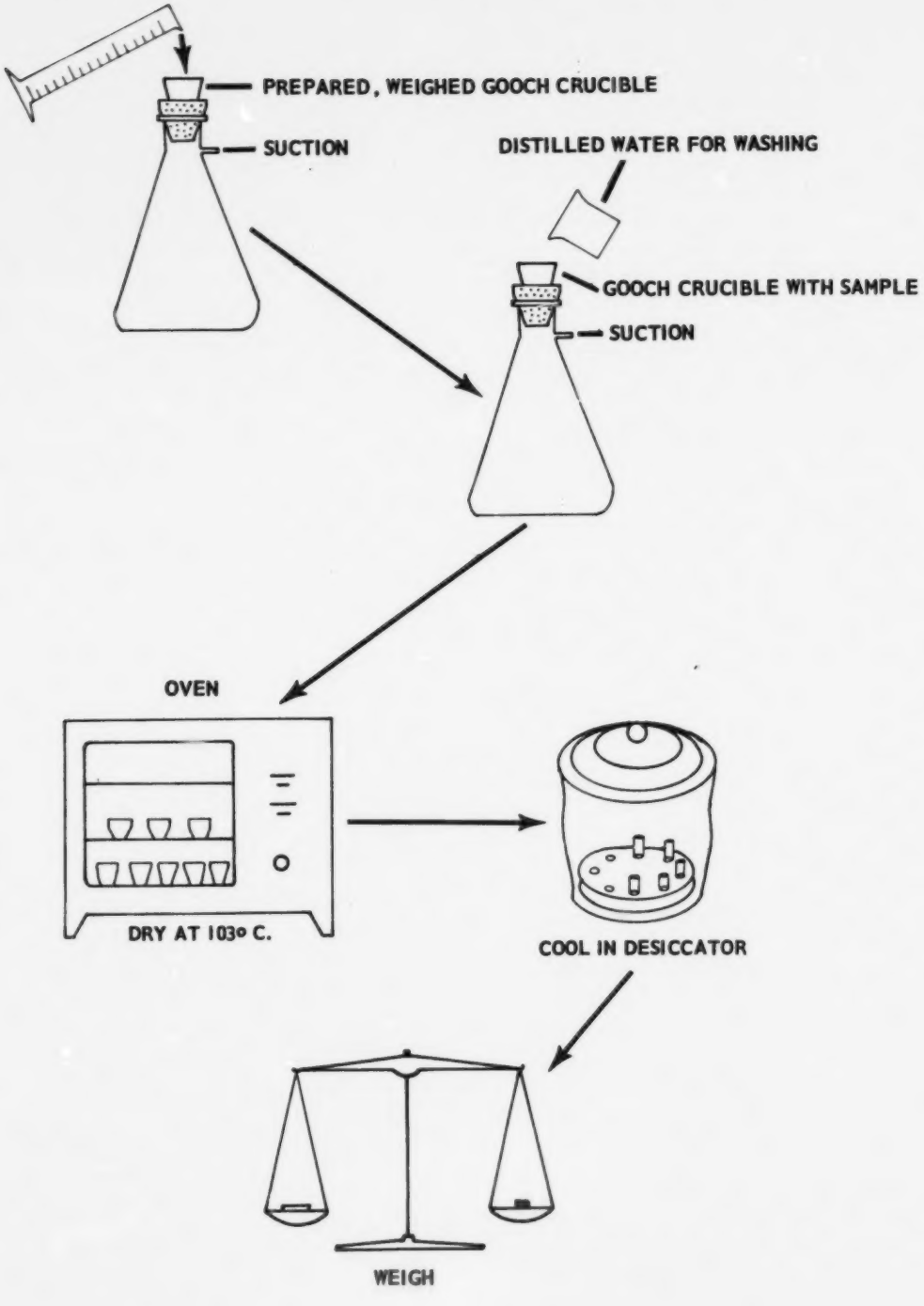
I. PREPARATION OF GOOCH CRUCIBLES



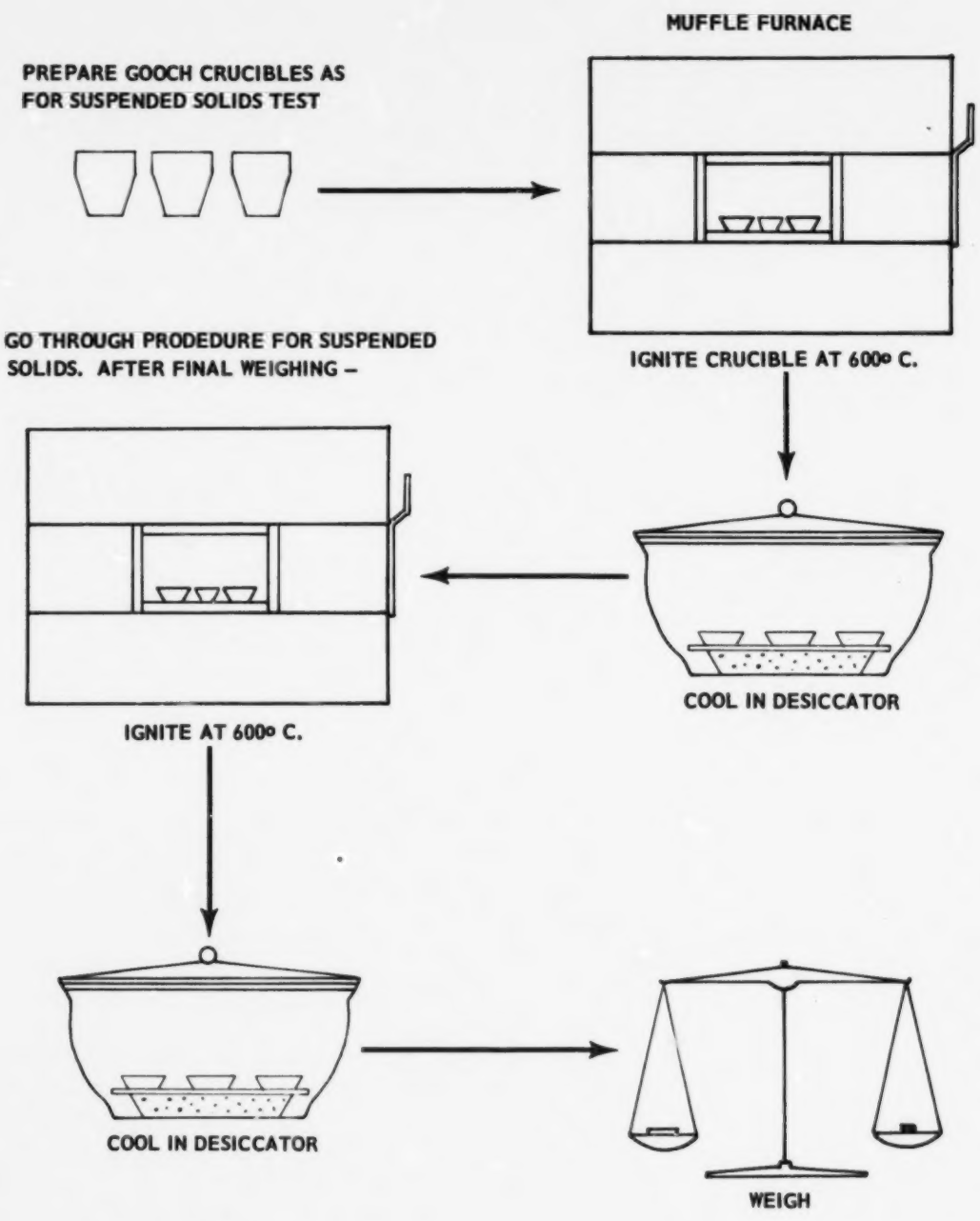
2. Treatment of Sample

Pour measured volume of sample into Gooch crucible

(See next page.)


PREPARED, WEIGHED GOOCH CRUCIBLE
SUCTION
DISTILLED WATER FOR WASHING
GOOCH CRUCIBLE WITH SAMPLE
SUCTION
OVEN
DRY AT 103° C.
COOL IN DESICCATOR
WEIGH

VOLATILE SUSPENDED SOLIDS TEST


MUFFLE FURNACE
PREPARE GOOCH CRUCIBLES AS FOR SUSPENDED SOLIDS TEST
IGNITE CRUCIBLE AT 600° C.
GO THROUGH PRODEDURE FOR SUSPENDED SOLIDS. AFTER FINAL WEIGHING –
COOL IN DESICCATOR
IGNITE AT 600° C.
COOL IN DESICCATOR
WEIGH

Recording of Results

1. Record weight of ignited dish or crucible.
2. Record weight of dish or crucible with sample (as in total or suspended solids determinations).
3. Record weight of ignited dish or crucible with sample.
4. Record volume of sample.

HOW TO MAKE THE TEST

1. Determine total solids in a pre-ignited evaporating dish or suspended solids in a pre-ignited Gooch crucible.
2. Ignite dish and sample at 600°C for 10-15 minutes, or until a white ash remains.
3. Cool in desiccator for 20-30 minutes.
4. Weigh and record weight.
5. Calculations.

(1) Volatile total solids

mg/l (or ppm) volatile total solids
= (weight in mg. of ignited dish with sample - weight in mg. of ignited dish with ignited sample) X $\frac{1000}{\text{ml of sample}}$

per cent (%) volatile total solids
= $\frac{\text{mg/l volatile total solids}}{\text{mg/l total solids}}$ X 100

(2) Volatile suspended solids

mg/l (or ppm) volatile suspended solids
= (weight in mg of ignited crucible with sample - weight in mg of ignited crucible with ignited sample) X $\frac{1000}{\text{ml of sample}}$

per cent (%) volatile suspended solids
= $\frac{\text{mg/l volatile suspended solids}}{\text{mg/l suspended solids}}$ X 100

(3) Fixed total solids

= total solids - volatile total solids

(4) Fixed suspended solids

= suspended solids - volatile suspended solids

SETTLEABLE SOLIDS IN ACTIVATED SLUDGE TEST

1. Fill Mallory Direct Reading Settlometer or any other large diameter graduated cylinder to 1000 cc/l mark with thoroughly mixed activated sludge.
2. Allow solids to settle quietly for 30 minutes.
3. Read the volume of solids in the bottom of the container.
4. Report the results as mls of settleable solids per liter.

SLUDGE VOLUME INDEX

Sludge volume index is defined as the volume in milliliters occupied by 1 gram of activated sludge.

$$\frac{\text{Settleable solids in ml per liter} \times 1000}{\text{mg/l suspended solids}} = \text{Sludge Volume Index}$$

SLUDGE DENSITY INDEX

Sludge density index is defined the reciprocal of the sludge volume index multiplied by 100.

$$\frac{1}{\text{Sludge Volume Index}} \times 100 = \text{Sludge Density Index}$$

SLUDGE AGE

In the activated sludge process, sludge age is defined as a measure of the length of time a particle of suspended solids has been undergoing aeration, expressed in days. It is usually computed by dividing the weight of the suspended solids in the aeration tank by the daily addition of new suspended solids having their origin in the raw waste. Assuming that no sludge blanket exists in the final clarifier:

$$\frac{\text{Volume of aeration tank (million gal.) x 8.34 x mg/l SS}}{\text{Settled sewage daily flow (million gal.) x 8.34 x (P.E.-F.E.) mg/l SS}} = \text{Sludge Age}$$

Where P.E. = primary effluent SS and F.E. = final effluent SS.

$$\frac{\text{Total pounds of activated sludge}}{\text{Total pounds SS removed from primary effluent per day}} = \text{Sludge Age}$$

SPECIFIC GRAVITY OF SEWAGE SLUDGE

A. REAGENTS AND APPARATUS:

1. Distilled water.
2. Trip scale or balance,
3. One wide mouth glass stoppered bottle or flask of about 8 oz. capacity or more.

B. PROCEDURE

1. Weigh the bottle or flask to the nearest 0.1 gram.
2. Fill to overflowing with distilled water, insert the stopper, dry with a cloth and weigh.
3. Completely empty the bottle, fill to overflowing with the well-mixed sludge and insert the stopper.
4. Wash the sludge from the outside of the bottle or flask, dry with a cloth and weigh.

C CALCULATIONS:

$$\frac{\text{Weight of the sludge}}{\text{Weight of distilled water}} = \text{Specific gravity of sludge}$$

Example:

Weight of bottle and distilled water	550.5 grams
Weight of bottle	250.5 grams
Weight of distilled water	300.0 grams

Weight of bottle and sludge	556.5 grams
Weight of bottle	250.5 grams
Weight of sludge	306.0 grams

$$\frac{306}{300} = 1.02 \text{ Specific gravity}$$

SLUDGE CONDITION FOR VACUUM FILTRATION

Grab a sample of ferric chloride and lime from the conditioning tanks. (These chemicals may be other than ferric chloride and lime).

1. Measure five 200 ml portions of sample into five 400 ml beakers. Number beakers 1 to 5.

2. Add 1.0, 2.0, 3.0, 4.0 and 5.0 mls of ferric chloride to the respective samples. Stir gently about 30 seconds.

3. Determine filtration time as described above.

4. Using that quantity of ferric chloride that requires three or four minutes to filter, repeat the test as follows.

5. Add the ferric chloride dose to each of five 200 ml samples. Stir 30 seconds and then add 1.0, 2.0, 3.0, 4.0 and 5.0 mls of the well-mixed lime solution to each sample. Stir and determine filtration time as above. pH value should not exceed 1.0.

6. Determine the optimum combination of ferric chloride and lime that will yield a filtration time of about two to three minutes. Less than one minute is better than necessary and more than four minutes is unsatisfactory.

CALCULATIONS

Ml of ferric chloride used = gallons ferric chloride per 200 gallons sludge.
Ml lime solution used = gallons lime solution per 200 gallons sludge.

AMMONIA NITROGEN TEST

A. REAGENTS

1. Ammonia free water
2. Permanent ammonia standards
3. Standard ammonium chloride
4. Nessler reagent
5. Sodium hydroxide, 12N
6. Copper sulfate solution, 10 percent

B. PROCEDURE

1. Place 100 ml. of the sample in a Nessler tube and add 1 ml. copper sulfate solution.
2. Mix by rotating and add 1 ml. of sodium hydroxide.
3. Mix again and allow to settle.
4. Pipette a measured portion of the clear supernatant liquor, 25 mls., depending upon the ammonia content, into a second Nessler tube and dilute to 100 ml. with ammonia free water.
5. If permanent ammonia standards are available, proceed to Step 6. If not, make up temporary standards by adding 0.2, 0.4, 0.6, 0.8, 1.0, 1.4, 1.7, 2.0, 2.5, 3.0 ml. of standard ammonia chloride to 100 ml. Nessler tubes and dilute to the mark with ammonia free water.
6. Add 2 ml. of Nessler reagent to the sample and to each temporary standard (if used).
7. After 10 minutes compare the colors and record the standard having a color nearest to that of the sample.

C. CALCULATIONS

a. Using permanent standards:

$$\frac{\text{mg } NH_3\text{-N in permanent standard} \times 1000}{\text{ml. portion used in step 4}} = \text{mg/l Ammonia nitrogen as N}$$

b. Using temporary standards:

$$\frac{\text{ml. } NH_4Cl \text{ in standard} \times 10}{\text{ml. portion used in step 4}} = \text{mg/l Ammonia nitrogen as N}$$

NITRITE NITROGEN TEST

A. REAGENTS

1. Aluminum hydroxide
2. Standard sodium nitrite
3. Sulfanilic acid
4. A - napthylamine

B. PROCEDURE - FILTER SAMPLE

1. If the sample is colored or turbid, clarify 150 ml by adding 2 ml of aluminum hydroxide.
2. Place a measured portion of the filtrate (10-50) ml, depending upon the nitrite content, into a 100 ml Nessler tube and make up to the mark with distilled water.
3. If permanent standards are available, proceed to Step 4; if not, make up temporary standards by adding 0.2, 0.4, 0.6, 0.8, 1.0, 2.0, or 2.5 ml of standard sodium nitrite in 100 ml Nessler tubes and make up to the mark with nitrite free water.
4. Add 2 ml of sulfanilic acid and 2 ml of a-naphthylamine to the sample and to each temporary standard if used.
5. Mix and allow to stand 10 minutes. Compare the colors and record the standard having a color nearest to that of the sample.

C. CALCULATIONS

a. Using permanent standards:

$$\frac{\text{mg } NO_2\text{-N in permanent standard} \times 1000}{\text{ml of sample}} = \text{mg/l Nitrite nitrogen as N}$$

b. Using temporary standard:

$$\frac{\text{ml standard } NaNO_2 \times 0.5}{\text{ml of sample}} = \text{mg/l Nitrite nitrogen as N}$$

NITRATE NITROGEN TEST

A. REAGENTS

1. Phenoldisulfonic acid
2. Sodium hydroxide, 12N
3. Standard nitrate solution

B. PROCEDURE

1. Determine chloride content of water using the CHLORIDES IN SEWAGE TEST found on page 52.
2. Remove the chlorides present by the CHLORIDES REMOVAL PROCEDURE found on page 72.
3. Filter 30-35 ml of sample through filter paper.
4. Evaporate 25 ml or the filtrate to dryness on a water bath, (use a smaller amount if nitrate content is high).
5. Moisten the residue with 1 ml of phenoldisulfonic acid.
6. Dilute to about 20 ml with distilled water.
7. Add 12N sodium hydroxide until the maximum yellow color is developed (not more than 5 to 6 ml or sodium hydroxide will be required).
8. Filter into a 100 ml Nessler tube and rinse the dish and paper with distilled water. Add the filtered rinsings to the filtrate and make up to the mark with distilled water.
9. If permanent standards are available, proceed to Step 8; if not, make up temporary standards by placing 0.2, 0.4, 0.6, 0.8, 1.0, 2.0, 3.0, 4.0, and 5.0 ml of standard sodium nitrate solution in 100 ml Nessler tubes and adding 2 ml of 50% sodium hydroxide.
10. Dilute to the mark with distilled water.
11. Compare the color and record the standard having a color nearest to that of the sample.

C. CALCULATIONS

a. Using permanent standards:

$$\frac{\text{mg } NO_3\text{-N in permanent standard} \times 1000}{\text{ml of sample in Step 2}} = \text{mg/l Nitrate nitrogen as N}$$

b. Using temporary standards:

$$\frac{\text{ml of standard } NaNO_3 \times 10}{\text{ml of sample in Step 2}} = \text{mg/l Nitrate nitrogen as N}$$

SULFITE TEST

1. Place 10 ml of 0.025N iodine and 5 ml of glacial acedic acid into each of two 250 ml Erlenmeyer flasks.

2. Add 100 ml of the freshly collected and cooled, but unfiltered, sample slowly and with constant mixing to one flask and 100 ml of distilled water to the other.

3. To the flask containing the sample add from a burette 0.025N sodium thiosulfate until the color of the iodine almost disappears. Add 1 ml of starch indicator and continue the addition of thiosulfate until the blue color just disappears. Record the ml of thiosulfate used.

CALCULATIONS

Let D = ml of thiosulfate used for distilled water
Let S = ml of thiosulfate used for sample
(D-S) x 0.91 = gpg sodium sulfite (Na_2SO_3)
To convert gpg to mg/l multiply by 17.1

SULFATE TEST

SULFATES - BENZIDINE METHOD

1. If the sample contains suspended matter, filter about 70 ml through a filter paper.

2. Measure 58.3 ml of the filtered sample into a 250 ml Erlenmeyer flask.

3. Add 10 ml of benzidine hydrochloride solution (2 per cent) and mix by giving the flask a whirling motion.

4. Allow the mixture to stand for about ten minutes.

5. Filter the precipitated benzidine sulfate onto a small filter paper. The solution should be refiltered through the same paper until filtrate is clear.

6. Add 1 ml of benzidine hydrochloride solution to the filtrate. If further precipitation takes place, filter through the same paper. Repeat the addition of benzidine sulfate until all of the sulfate is precipitated and removed to the paper.

7. Wash the flask and precipitate on the paper with several small portions of distilled water. Allow each portion to drain through the paper before the next is added.

8. Transfer the paper containing the benzidine sulfate to the original flask, add about 25 ml of distilled water and two drops of phenolphthalein indicator.

9. Add 0.143N (N/7) sodium hydroxide from a burette until the first permanent pink color is obtained. Be sure that the paper is completely disintegrated and that the color is permanent.

10. Record the ml sodium hydroxide used.

CALCULATIONS

Ml of NaOH x 10 = gpg SO_4 as sodium sulfate (Na_2SO_4)
Ml of NaOH x 6.32 = gpg SO_4
To convert gpg to mg/l multiply by 17.1

CARBON DIOXIDE TEST

1. Fill a 100 ml Nessler tube to the mark with the sample.

2. Add 10 drops of phenolphthalein indicator.

3. Add N/44 sodium hydroxide from a burette, stirring gently, until a slight permanent pink color appears. Record the number of ml of sodium hydroxide used.

CALCULATIONS

Ml of N/44 NaOH X 10 = mg/l CO_2
Test should be made at time the sample is collected. If the sample has a high CO_2 content, about 3/4 of the NaOH required should be added to the beaker before adding the sample.

HYDROGEN SULFIDE TEST

A. EQUIPMENT NEEDED

1. One 1000 ml capacity graduated cylinder.
2. Two 250-500 ml capacity Erlenmeyer flasks.
3. Pipette
4. Siphon

B. CHEMICALS NEEDED

1. 0.025 N iodine solution
2. Potassium iodide crystals
3. 0.025 N sodium thiosulfate
4. Starch indicator

C. METHOD

1. Siphon 500 ml of the sample into a graduated cylinder.
2. Pipette 10 ml of the 0.025 N iodine solution into each of two Erlenmeyer flasks.
3. Add about 1 gram of potassium iodide crystals.
4. Add 200 ml of distilled water to one flask.
5. Siphon 200 ml of sample from graduate into other flask.
6. Titrate both the distilled water blank and the sample with 0.25N Sodium thiosulfate using starch as an indicator near end of titration. Record ml of thiosulfate used.

D. CALCULATIONS

Let x = ml of Sodium thiosulfate used for sample
Let y = ml of Sodium thiosulfate used for distilled water

$$\frac{(y-x) \times 426}{\text{ml of sample}} = \text{mg/l of Hydrogen sulfide}$$

CHLORIDES IN SEWAGE TEST

REAGENTS AND APPARATUS

1. Standard silver nitrate solution (1 ml equivalent of 0.5 mg chloride ion)
2. Potassium chromate indicator - 5 per cent solution
3. Chloride free sodium bicarbonate
4. 25 ml burette
5. 200 ml Erlenmeyer flask or porcelain casserole

PROCEDURE

1. Pipette 50 or 100 mls of the sample into the flask or casserole, depending upon the chloride content.
2. Add 1 ml potassium chromate indicator.

3. Titrate to first permanent red color with standard silver nitrate. If more than 7 or 8 mls of silver nitrate are required, repeat entire procedure, using a smaller sample diluted to 50 mls with chloride free distilled water.

4. Calculate chloride content as follows:

$$\frac{(\text{ml silver nitrate} - \text{blank}) \times 500}{\text{ml of sample}} = \text{mg/l chloride}$$

PHOSPHATE TEST

Phosphates are usually found in wastewater. Detergents contain phosphates and polyphosphates may be present in addition to the usual orthophosphate.

For differentiation of ortho and polyphosphates, consult "Standard Methods" and "FWPCA Methods For Chemical Analysis Of Water and Wastes," November, 1969.

Color comparators are available for making phosphate analyses. These analyses are satisfactory for field work and operation control analyses.

ALKALINITY SEWAGE TEST

NOTE: The alkalinity determination may be performed more accurately using the Potentiometric Titration Method given for the ALKALINITY SLUDGE TEST.

1. Pipette 100 mls of the sample in an Erlenmeyer flask or beaker.

2. Add three drops of phenophthalein indicator to the sample.

3. If the sample becomes pink, add 0.02N sulfuric acid from a burette until the pink color just disappears and record the number of mls of acid used.

4. Add 3 drops of methyl orange indicator to the sample.

5. If the sample becomes yellow, add 0.02N sulfuric acid until the first difference in color is noted. The end point is orange. Record the mls of acid used.

CALCULATIONS

Total alkalinity as mg/l $CaCO_3$ = total mls acid used x 10
Hydroxide (OH) - normal carbonate (CO_3) - and bicarbonate (HCO_3) are determined below.

P = ml of 0.02N sulfuric acid used for the titration with phenolphthalein
T = ml of acid used for total titration (phenolphthalein plus methyl orange)

There are five possible conditions:

1. P = T
   Hydroxide mg/l = P x 10

2. P > 1/2 T
   Hydroxide mg/l = (2P - T) x 10
   Normal carbonate mg/l = 2(T - P) x 10

3. P = 1/2 T
   Normal carbonate mg/l = T x 10

4. P < 1/2 T
   Normal carbonate mg/l = 2P x 10
   Bicarbonate mg/l = (T - 2P) x 10

5. P = 0
   Bicarbonate mg/l = T x 10

All the above results are in terms of mg/l as $CaCO_3$.

ALKALINITY SLUDGE TEST

(Potentiometric Titration Method)

APPARATUS

Use a commercial instrument for measuring pH with a glass electrode. Adjust meter with a 7.0 pH buffer.

1. Measure 100 ml of the settled sample and pour into a beaker.

2. Measure the pH.

3. If the pH is above 8.3 the sample contains hydroxide or normal carbonate alkalinity.

4. Titrate with 0.02N sulfuric acid stirring the sample during titration. When the meter reads 8.3 record mls acid used.

5. Continue to titrate with 0.02N sulfuric acid until meter reads 4.5. Record mls acid used.

CALCULATIONS

Total alkalinity as mg/l $CaCO_3$ = total mls acid used x 10
Hydroxide (OH) - normal carbonate (CO_3) - and bicarbonate (HCO_3) are determined below.

P = ml of 0.02N sulfuric acid used for the titration to pH 8.3
T = ml of acid used for total titration (above pH 8.3 plus acid used to 4.5)

There are five possible conditions:

1. P = T
   Hydroxide mg/l = P x 10

2. P > 1/2 T
   Hydroxide mg/l = (2P - T) x 10
   Normal carbonate mg/l = 2(T - P) x 10

3. P = 1/2 T
   Normal carbonate mg/l = T x 10

4. P < 1/2 T
   Normal carbonate mg/l = 2P x 10
   Bicarbonate mg/l = (T - 2P) x 10

5. P = 0
   Bicarbonate mg/l = T x 10

All of the above results are in terms of mg/l as $CaCO_3$.

ACIDITY SEWAGE TEST

1. Pipette 100 mls of the sample into an Erlenmeyer flask or beaker.

2. Add 3 drops of phenolphthalein indicator.

3. Add 0.02N sodium hydroxide from a burette until the first permanent pink color appears and record the number of mls of sodium hydroxide used.

CALCULATIONS

Ml of 0.02N NaOH x 10 = mg/l total acidity expressed in terms of $CaCO_3$

ACIDITY SLUDGE TEST

APPARATUS

Use a commercial instrument for measuring pH with a glass electrode. Adjust meter to a pH of 7.0.

1. Measure 100 mls of the settled sample and pour into a beaker.

2. Measure the pH.

3. Add 0.02N sodium hydroxide from a burette until the pH meter reads 8.3. Record number of mls of sodium hydroxide used.

CALCULATIONS

Ml of 0.02N NaOH x 10 = mg/l total acidity expressed in terms of $CaCO_3$.

CHLORINE DEMAND AND STANDARD SOLUTIONS

1. Chlorine demand of a water must be satisfied before a residual can be produced. The materials causing the chlorine demand are: bacteria, organic matter, and some minerals.

2. Chlorination of water and sewage for sterilization and odor control is being practiced in many treatment plants. To calculate the dosage, the demand will have to be known. There are a number of methods used for the chlorine demand determination. The one used in this manual is simple and accurate. This method uses a series of samples treated with varying amounts of chlorine. The sample with the least amount of added chlorine, which shows a residual, is used in calculating the chlorine demand.

3. The chlorine demand of sewage varies widely from hour to hour. This variation is greater for raw and settled sewage than for final effluents. The chlorine demand of raw sewage is usually greater in warm weather than in winter. There is a close relationship between the chlorine demand and the oxygen demand of sewage.

4. The greatest benefit from the use of chlorine in sewage works operation is for disinfection of the plant effluent. A dosage sufficient to produce a residual of 0.5 mg/l after 30 minutes contact time should be maintained. The purpose of chlorination is to destroy harmful bacteria. Other uses may be made of chlorine as it is a strong oxidizing agent.

5. Bleach is often used for preparing solutions for the chlorine demand test.

SOLUTION FOR CHLORINE DEMAND TEST

Pipette exactly 20 mls of "purex" (5% chlorine solution) into a one liter volumetric flask and fill to the mark with distilled water. This solution will contain 1000 mg/l of chlorine.

1 ml of solution in 1 liter of sample is equal to 1 mg/l.

1. Measure 250 mls of the well-mixed sewage to be tested into a series of eight 300 ml capacity beakers.

2. Add 0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.7, 0.8 mls of the chlorine solution to the beakers in succession.

3. Mix each beaker by gently shaking and allow to stand for 30 minutes.

4. Add a crystal of potassium iodide and 1 ml of starch solution to each beaker and mix.

5. Record the mls of chlorine water in the beaker containing the least amount of chlorine water which shows a blue color.

6. Ml of chlorine water in first bottle to show a blue color x 4 = mg/l chlorine demand.

JAR TEST FOR BLUE GREEN ALGAE CONTROL

(Amount of HTH to Control Blue Green Algae)

Pipette exactly 20 mls of "purex" (5% chlorine solution) into a one liter volumetric flask and fill to the mark with distilled water. This solution will contain 1000 mg/l of chlorine.

One ml of solution added to one liter of sample is equal to one mg/l.

Measure 250 mls of the well-mixed sewage to be tested into a series of eight 300 ml capacity beakers. Quart jars can be substituted for the beakers. Add 1.0, 1.5, 2.0, 2.5, 3.0, 3.5, 4.0 and 4.5 of the chlorine solution to the beakers in succession.

Mix each beaker gently shaking and allow to stand for one hour.

Observe the containers. The first one indicating the algae has been bleached out is the correct dosage.

$$\text{Pounds HTH per acre lagoon 3-foot depth} = \frac{\text{Milliliter chlorine water applied to jar} \times 8.34 \times 4}{70}$$

EXAMPLE: Assume jar No. 5 indicated the algae had been bleached.

$$\text{Pounds HTH per acre 3-foot depth} = \frac{3 \times 8.34 \times 4}{70} = 143 \text{ pounds}$$

OIL AND GREASE TEST

1. Determine the tare weight of a clean and dried 125 ml E-flask.

2. Place 500 mls of sample in a 1000 mls separatory funnel. Add 1.25 ml of concentrated H_2SO_4 to the sample. Rinse the sample container with 15 mls of Petroleum Ether (30°-60°C).

3. Add 25 mls of additional Petroleum Ether to the separatory funnel, shake, and allow the ether layer to separate out. Drain out the H_2O phase into another separatory funnel and filter the ether phase through anhydrous sodium sulfate on Phase Separating Paper, PSP, (Silicone Treated) Whatman 1^{PS} 9.0 cm type into the 125 ml E-flask.

4. Rinse out the first separatory funnel with 15 mls of ether and add this to the second separatory funnel. Add 50 mls of ether, shake and extract the ether layer as done before.

5. Place the combined ether extracts in the 125 ml flask. Put them into a water bath at $70^{o}C$. Allow them to evaporate to dryness.

6. Reweigh the 125 ml E-flask.

7. Calculation:

   mg/l Total Oil and Grease = (gross-tare weight) X 2.

GREASE (SOXHLET EXTRACTION METHOD) TEST

1. In a wide-mouth bottle collect a sample of sewage of measured volume to contain from 50 to 150 mg grease.

2. Acidity to pH 1.0 usually 3 ml concentrated hydrochloric acid per liter is sufficient.

3. In a 12 cm Buchner funnel prepare a filter consisting of a muslin cloth disc overlaid with filter paper. Wet the paper and muslin and press down the edges of paper. Filter with vacuum 100 ml filter aid suspension through the cloth and paper. Wash with one liter of distilled water. Apply vacuum until no more water passes through the filter.

4. Filter the acidified sample through the prepared filter. Apply vacuum until no water passes through the filter.

5. Remove the filter paper to a watch-glass by means of forceps, adding the materials adhering to the edges of the muslin cloth disc; wipe the collecting vessel, the stirring rod, and Buchner funnel with filter paper to remove all grease and solids materials. Add the filter paper to that on the watch-glass and roll them together and fit in a paper extraction thimble.

6. Dry at 103°C - 30 minutes.

7. Weigh extraction flask and extract grease in a Soxhlet apparatus using petroleum ether at a rate of 20 cycles per hour for 4 hours.

8. Distill ether from the extraction flask in a water bath at 70°C.

9. Dry by placing the flask on a steam bath and draw air through the flask by means of vacuum applied for 15 minutes.

10. Cool in a desiccator one-half hour and weigh.

CALCULATIONS

$$\text{Total grease mg/l} = \frac{\text{mg increase in weight of flask} \times 1000}{\text{ml sample}}$$

pH OF SEWAGE SLUDGE — COLORIMETRIC METHOD

HYDROGEN ION CONCENTRATION (pH) OF SEWAGE SLUDGE

COLORIMETRIC METHOD

1. Place about 20 ml of the sludge in a 100 ml graduate and dilute with distilled water to 100 ml mark.

2. Mix well and settle. (The sludge may be clarified by centrifuging instead of using the procedure given in Steps 1 and 2.)

3. Place 10 ml of supernatant liquor into each of the two or three tubes provided with the pH apparatus.

4. To one tube add the correct amount of indicator.

5. Place the tubes in the comparator in such a manner that the color standards are opposite the tubes not containing the indicator. The color comparison must be made by looking through the same thickness of liquid having the same color and turbidity as the sample.

6. Compare the colors and select the standard having a color nearest to that of the sample.

HYDROGEN ION CONCENTRATION (pH) OF SEWAGE

pH OF SEWAGE — COLORIMETRIC METHOD

1. Place 10 mls of sample into each of the two or three tubes provided with the pH apparatus.

2. To one tube add the correct amount of indicator.

3. Place the tubes in the comparator in such a manner that the color standards are opposite the tubes not containing the indicator. The color comparison must be made by looking through the same thickness of liquid having the same color and turbidity of sample.

4. Compare the colors and select the standard having a color nearest to that of the sample.

HYDROGEN-ION CONCENTRATION DISCUSSION

The acid or alkali intensity, hydrogen-ion concentration, of a solution is found by determining the pH.

Ions are electrically charged atoms or groups of atoms. When acid base or salt is dissolved in a suitable solvent the molecules dissociate into smaller units, some of which have a positive electric charge and others are equal negative charge. For example:

Hydrochloric acid dissociates into positively charged hydrogen ions and negatively charged chlorine ions. $HCl \rightleftarrows H^+ + Cl^-$

Water dissociates into positively charged hydrogen ions and negatively charged hydroxyl ions. $HOH \rightleftarrows H^+ + OH^-$

It has been determined that there are 1/10,000,000 grams of hydrogen ions and the same quantity of hydroxyl ions in one liter of pure water.

The product of the H and OH ions equal a constant value. Therefore, if the concentration of H ions is increased there is a corresponding decrease in OH ions. For example:

If the concentration of H ions is increased from 1/10,000,000 (10^{-7}) to 1/100,000 (10^{-5}) then the OH ions are decreased from 1/10,000,000 or (10^{-7}) 1/1,000,000 (10^{-9}) gram.

The acidity or alkalinity, hydrogen-ion concentration, of a solution is given in terms of the pH. For convenience the negative exponent of the hydrogen ion concentration is used to express the amount of hydrogen ions present: For example:

If a solution has a hydrogen-ion concentration of 1/100,000 or 10^{-5}, the pH value is 5.0.

The pH scale extends from 0 to 14 with the neutral point at 7.0. As the pH value decreases the hydrogen-ion concentration increases, and vice versa. A change in pH value is not in direct proportion to the numerical values. A change of 1 in pH value means the hydrogen ion concentration has changed 10 times. A change of 2, 100 times etc.

pH value may be determined colorimetrically. The color of certain dyes change as the pH value of the solution changes. Indicators available for use and their effective range in determining pH value are as follows:

INDICATORS FOR pH DETERMINATION

NAME	pH RANGE	COLOR CHANGE
Methyl Red	4.4 to 6.0	Red to Yellow
Brom Cresol Purple	5.2 to 6.8	Yellow to Purple
Brom Thymol Blue	6.0 to 7.6	Yellow to Blue
Phenol Red	6.8 to 8.4	Yellow to Red

NAME	pH RANGE	COLOR CHANGE
Cresol Red	7.2 to 8.8	Amber to Red
Thymol Blue	8.0 to 9.6	Yellow to Blue

Color comparators can be purchased for making this test. Electric pH meters can also be purchased for making this test. Determination of pH with an electric pH meter may be more accurate than with colorimetric methods.

ACIDS — VOLATILE

This test is useful for early detection of malfunction in digesters. The distillation procedures are cumbersome and give results varying with the type of apparatus, composition of the acids, distillation rate, and amount distilled. In this method, no distillation is necessary. The volatile acids are titrated directly after removal of the bicarbonate ions as carbon dioxide.

APPARATUS AND REAGENTS

Buffer solution, 4.00 and 7.00
Standard 0.02N sulfuric acid solution (H_2SO_4)
Standard 0.02N sodium hydroxide (NaOH)
25 ml pipette or graduate to measure sample
10 ml graduated pipette or burette for titrating
250 ml beaker
Adjustable hot plate
Electronic pH meter

PROCEDURE

1. Set the pH meter at 7.0 using the 7.0 pH buffer or centrifuging sample.

2. Measure a known quantity of clear sample into a 250 ml beaker or Erlenmeyer flask and insert the glass electrode of the pH meter. Usually 25 ml is sufficient, but smaller amounts may be advisable if the alkalinity is high.

3. Titrate with .02N sulfuric acid to pH 4.0 while swirling the contents of the beaker. The glass electrode should be held firmly against the side of the beaker during this procedure to prevent breakage. Record this value as alkalinity. Continue titrating to pH 3.5 to 3.3.

4. Carefully set the pH meter by using a 4.00 pH buffer while lightly boiling the sample a minimum of 3 minutes. Cool in cold water to original temperature.

5. Titrate sample with standard 0.02N sodium hydroxide up to pH 4.00 and note reading. Continue titrating to pH 7.0 and note readings.

6. Calculate volatile acid alkalinity (volume required in #5 to go from pH 4.0 to pH 7.0).

$$\text{Volatile Acid Alkalinity} = \frac{\text{ml } 0.02N \text{ NaOH} \times 1000}{\text{ml sample}}$$

7. Calculate Volatile Acids
   Volatile Acids = Volatile acid alkalinity (when this value is less than 180 mg/1)

   Volatile Acids = 1.5 x Volatile Acid Alkalinity (when this value is more than 180 mg/1)

CARBON DIOXIDE IN SEWAGE GAS

1. Waste a portion of the gas to the air in order to clear the lines and to obtain a representative sample. If the gas is not piped to the laboratory, a sample may be collected at any convenient place on the gas domes or from the lines to the burners. It should be collected in a flat rubber gas bag capable of holding about 1 liter.

2. Raise the leveling tube and fill the measuring pipette completely with the liquid (mercury is preferred).

3. Attach the bag or pipe line and draw about 100 mls of the gas into the pipette by lowering the leveling tube.

4. Close the stopcock connecting the gas bag or gas line and carefully measure the volume of gas in the pipette. Let this volume in ml = A. (The volume of gas in the pipette should always be measured by holding the level of the liquid in the leveling tube at the same elevation as that in the pipette.)

5. Open the connection to the potassium hydroxide (100 grams dissolved in 200 mls distilled water) pipette and pass the gas into the pipette, allowing it to remain in contact with the solution for some time.

6. By lowering the leveling tube, bring back the entire volume of remaining gas into the measuring pipette.

7. Close the connection and measure the volume as before.

8. Repeat steps 5, 6 and 7 until there is no further gas absorbed from contact with the potassium hydroxide solution.

NOTE - The apparatus must be free from leaks. Keep the glass stopcocks well greased.

CALCULATIONS

$$\frac{\text{ml of gas absorbed X 100}}{\text{A}} = \text{per cent carbon dioxide}$$

Gas analysis equipment can be obtained from companies supplying laboratory equipment. They are easier for the operator to use for making gas analysis.

HYDROGEN, METHANE AND B.T.U. IN SEWAGE GAS

1. Record the volume of gas remaining in the measuring pipette from the carbon dioxide determination. Let the volume in mls = B.

2. Discard all but 10 mls of this gas.

3. Lower the leveling tube and open the stopcock to the air, drawing in air until the volume is about 95 to 100 mls.

4. Measure accurately the volume in mls of the mixture.

5. Allow the gases to mix thoroughly.

6. Close the stopcock and the clamp on the leveling tube connection and explode or burn the gas in the pipette.

7. Allow the gas to cool to room temperature, open the check on the leveling tube and read the volume in mls of gas remaining in the pipette.

8. Determine the amount of carbon dioxide produced by passing the gases into the potassium hydroxide pipette several times until no further loss in volume is obtained.

9. Again read the volume of gas in measuring pipette.
Let mls in step No. 4 - mls in step No. 7 = C.
Let mls in step No. 7 - mls in step No. 9 = D.

CALCULATIONS

$\frac{10BD}{A}$ = per cent methane

$\frac{6.67\ B\ (C - 2D)}{A}$ = per cent hydrogen

(Per cent methane X 10.03) + (per cent hydrogen X 3.29) = B.T.U. per cubic foot (high heat value, 62°F and 760 mm)

(Per cent methane X 9.13) + (per cent hydrogen X 2.81) = B.T.U. per cubic foot (low heat value, 62°F and 760 mm)

Gas analysis equipment can be obtained from companies supplying laboratory equipment. They are easier for the operator to use for making gas analysis.

BACTERIAL EXAMINATION

(Membrane Filter Method)

SAMPLE COLLECTION

Take the sample with top of the bottle upstream or into flow; if no flow, use a sweeping motion moving top of bottle away from hand. Fill bottle only about two-thirds full to allow room for shaking. The hand must be kept away from the mouth of the bottle.

PREPARATION OF MEDIA AND REAGENTS

Buffered Dilution Water

Stock Phosphate Buffer Solution

Dissolve 34 grams potassium dihydrogen phosphate (KH_2PO_4) in 500 mls distilled water, adjust to pH 7.2 with 1N NaOH and make up to 1 liter with distilled water. Add 1.25 mls stock phosphate buffer to each liter of distilled water used for dilution bottles, rinse water and dilution water used in filter apparatus. Autoclave at 121°C at 15 psi for 20 minutes.

Dilution Bottle Preparation

The bottle should be filled with the proper amount of the buffered dilution water so that after autoclaving the volume is 99 mls $\pm$ 2 mls.

M-Endo Media

To each 980 mls distilled water add 20 mls 95% ethel alcohol and dissolve 48 grams dehydrated media. Place flask containing media in water bath, bring media just to the boiling point. Dispense 1.8 to 2.0 mls to each dish with pad to be used (make sure pad is saturated).

M.F.C. Broth Media

Dissolve 3.7 grams dehydrated M.F.C. Broth Base in 100 mls distilled water. Add one ml 1% rosolic acid solution. Place flask containing media in water bath, heat media to boiling, cool to room temperature, and add about 2 mls to each dish and pad to be used.

(Rosolic Acid Preparation - Dissolve one gram rosolic acid in 100 mls. 0.2N sodium hydroxide)

K.F. Streptococcus Agar

Dissolve 7.6 grams dehydrated media in 100 mls distilled water in sterile flask with aluminum foil cover. Place the flask in a boiling water bath, melt the dehydrated medium and leave in the boiling water bath an additional 5 minutes. Cool the medium to 50-60°C, add 1.0 ml of TPTC reagent and mix. Pour 5-8 mls to each 50 mm dish.

(TPTC - 2,3, 5 triphenyl tetrazolium chloride is prepared by adding one gram to 100 mls distilled water bringing to a boil. Store in screw-capped tube in refrigerator until use.)

PREPARATION OF APPARATUS

Sample Bottles

Autoclave at 121°C at 15 pounds pressure for 20 minutes.

Pipettes

Sterilize in oven at 180°C for 2 hours in pipette cans, or autoclave at 121°C at 15 psi for 20 minutes.

Filter Apparatus

Autoclave at 121°C at 15 pounds pressure for 20 minutes, or use ultraviolet light. The funnel need not be sterilized between samples of low bacterial density, but should be rinsed well. Where samples are highly contaminated, apparatus should be sterilized between each sample either by boiling or ultraviolet light.

Forceps

Sterilize between each operation by dipping in ethyl alcohol and burning off.

TOTAL COLIFORM

EQUIPMENT AND REAGENTS NEEDED

Balance (sensitivity .1 gram)
Filtration apparatus
Filters (grided) and pads (47 mm size with a pour size of 0.45 microns)
Forceps
Bunsen burner or alcohol lamp for sterilizing forceps
Petri dishes (50 mm size)
M-Endo media (Difco, Baltimore Biological Laboratory, etc.)
Vacuum flask
Source of vacuum (pump, vacuum line, water asperator, etc.) 15 psi maximum vacuum
Ethyl alcohol (not denatured)
Standard dilution bottles
Pipettes 1.0 and 1.1 ml
10 and 11 ml wide bore
10 ml serological
Pipette cans (aluminum or stainless steel)
Sterilizer (autoclave or pressure cooker)
Hot air oven (200°C)
Source of suitable distilled water
Sample bottles (100 to 200 ml size wide mouth) autoclave
Erlenmeyer flasks (100 mls or more depending on amount of media needed at a time)
Graduated cylinders (100 mls, 250 mls, and 1000 mls)
Erlenmeyer flask screw-capped (leter size or other convenient size for storing sterilized rinse water)
Water bath for heating media
Gas burner or hot plate for heat source
Hand-tally
Fluorescent light in housing permitting placement close to and as directly as possible over membrane filter for counting
Optical assistance in counting colonies (preferred wide field binocular microscope 10 times or 15 times) (less desirable simple lens with magnification of 5 times)

PROCEDURE

1. Select dilution range that is expected to give a 20 to 80 plate count, then plate one dilution above and one dilution below making a total of three dilutions. When no information is known about the sample, more dilutions may be needed. Where there have been several samples analyzed at a given point, less dilutions may be used. See appendix.

2. Place filter on filter apparatus.

3. If 20 mls or less of sample is to be filtered, add 20 mls of sterilized buffered dilution water to funnel, then add the sample and filter. If more than 20 mls of sample is used, add directly to funnel and filter.

4. Rinse funnel with sterilized dilution water and draw through filter.

5. Remove filter and place on M-Endo pad in dish.

6. Place in incubator inverted for 24 hours $\pm$ 2 hours at 35°C $\pm$ 0.5°C.

7. Remove from incubator and count all colonies that develop metallic sheen.

8. Record number of colonies and report number per 100 mls of sample. See appendix.

FECAL COLIFORM

EQUIPMENT AND REAGENTS NEEDED

Same as Total Coliform except:

Roslic acid
M.F.C. broth base in place of M-Endo media
Plastic bags (water tight)

PROCEDURE

1. Same except plate should be 20-60 colonies. See appendix.

2. Place filter on filter apparatus.

3. If 20 mls or less of sample is to be filtered, add 20 mls of sterilized buffered dilution water to funnel, then add the sample and filter. If more than 20 mls of sample is used, add directly to funnel and filter.

4. Rinse funnel with sterilized dilution water and draw through filter.

5. Place on pad saturated with M.F.C. broth media.

6. Incubate inverted in water bath at 44.5°C for 22 hours ± 2 hours.

7. Count all blue colonies that develop.

8. Record the number and report the count per 100 mls of sample. See appendix.

FECAL STREPTOCOCCUS

EQUIPMENT AND REAGENTS NEEDED

Same as Total Coliform except:

K.F. Streptococcus agar in place of M-Endo media

PROCEDURE

1. Same except plate count should be 20-100 colonies. See appendix.

2. Place filter on filter apparatus.

3. If 20 mls or less of sample is to be filtered, add 20 mls of sterilized buffered dilution water to funnel, then add the sample and filter. If more than 20 mls of sample is used, add directly to funnel and filter.

4. Rinse funnel with sterilized dilution water and draw through filter.

5. Place filter on the K.F. Streptococcus agar.

6. Incubate inverted at 35°C ± 0.5°C for 48 hours.

7. Count all colonies that develop a pink to dark wine color.

8. Record number and report count per 100 mls of sample. See appendix.

CHLORIDE REMOVAL PROCEDURE

A. REAGENTS

1. Standard silver sulfate solution:

   Dissolve 4.40 grams $Ag_2 SO_4$, free from nitrate, in distilled water and dilute to 1.0 liter.

B. PROCEDURE

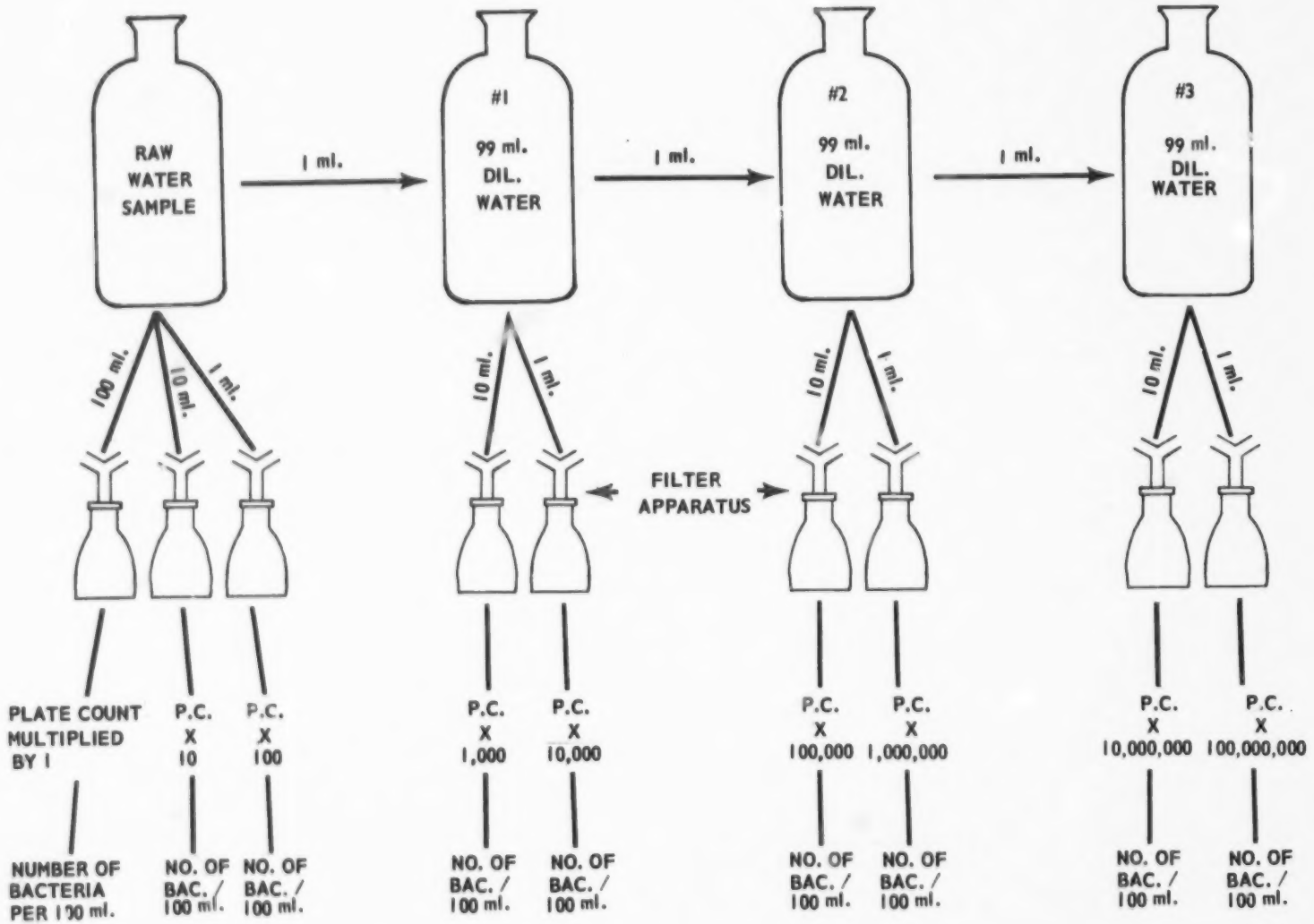
1. Determine chloride content of the water.

2. Treat 100 ml of sample with standard silver sulfate solution; 1.00 ml of standard silver sulfate solution should be added for each mg of chloride found in step 1.

3. Remove the precipitated chloride by either filtration or by centrifugation. Formation of the precipitate may be aided by heating the solution.

APPENDIX — BACTERIAL SAMPLING DILUTION PROCEDURE

(Membrane Filter Method)



CONVERSION FACTORS

for Operators

The following factors have been extracted from "Conversion Factors for Engineers" with permission of Dorr Oliver, Inc.

MULTIPLY	BY	TO OBTAIN
Acres	43,560	Square feet
Acre-feet	43,560	Cubic feet
Acre-feet	325,851	Gallons
Centimeters	0.3937	Inches
Cubic feet	1728	Cubic inches
Cubic feet	7.48052	Gallons
Cubic feet	28.32	Liters
Cubic feet/second	448.831	Gallons/minute
Cubic feet/second	0.646317	Million gallons/day
Cubic yards	27	Cubic feet
Degrees (angle)	60	Minutes
Feet	30.48	Centimeters
Feet	12	Inches
Feet	0.3048	Meters
Feet	1/3	Yards
Feet of water	0.4335	Pounds/square inch
Gallons	0.1337	Cubic feet
Gallons	3.785	Liters
Gallons	8	Pints (liq.)
Gallons	4	Quarts (liq.)
Gallons, Imperial	1.20095	U.S. gallons
Gallons U.S.	0.83267	Imperial gallons
Gallons water	8.3453	Pounds of water
Gallons/min.	2.228×10^{-3}	Cubic feet/sec.
Gallons/min.	8.0208/area (sq. ft.)	Overflow rate (ft/hr)
Grains/U.S. gal.	17.118	Parts/million
Grains/U.S. gal.	142.86	Lbs./million gal.
Grams	0.03527	Ounces
Grams	2.205×10^{-3}	Pounds
Grams/Liter	58.417	Grains/gal.

CONVERSION FACTORS (Continued)

MULTIPLY	BY	TO OBTAIN
Grams/liter	1000	Parts/million (approx.)
Horse-power	33,000	Foot-lbs/min.
Horse-power	0.7457	Kilowatts
Horse-power	745.7	Watts
Inches	2.540	Centimeters
Inches of mercury	1.133	Feet of water
Inches of mercury	0.4912	Lbs./sq. inch
Inches of water	0.07355	Inches of mercury
Inches of water	0.03613	Lbs./sq. inch
Kilowatt-hours	1.341	Horse-power-hrs.
Liters	0.03531	Cubic feet
Liters	0.2642	Gallons
Liters	1.057	Quarts (liq.)
$\frac{\text{Width (in)} \times \text{Thickness (in)}}{12}$	Length (ft)	Board feet
Meters	3.281	Feet
Meters	39.37	Inches
Miles	5280	Feet
Miles	1760	Yards
Milligrams/liter	1	Parts/million (approx.)
Million gals./day	1.54723	Cubic ft./sec.
Ounces	0.0625	Pounds
Ounces	28.349527	Grams
Overflow rate (ft/hr)	0.12468 x area $\frac{\text{sq ft}}{}$	Gals./min.
Parts/million	0.0584	Grains/U.S. gal.
Parts/million	8.345	Lbs./million gal.
Pounds	16	Ounces
Pounds	7000	Grains
Pounds	453.5924	Grams
Pounds of water	0.01602	Cubic feet
Pounds of water	0.1198	Gallons
Pounds/sq. inch	2.307	Feet of water
Pounds/sq. inch	2.036	Inches of mercury
Revolutions	360	Degrees
Square feet	2.296×10^{-5}	Acres
Square feet	144	Square inches

CONVERSION FACTORS (Continued)

MULTIPLY	BY	TO OBTAIN
Square feet	1/9	Square yards
Square inches	6.542	Square centimeters
Square meters	10.76	Square feet
Square miles	640	Acres
Square yards	9	Square feet
Temp. (°C) + 17.78	1.8	Temp. (°F)
Temp. (°F) - 32	5/9	Temp. (°C)
Watts	1.341×10^{-3}	Horse-power
Yards	3	Feet
Yards	36	Inches
Yards	0.9144	Meters

UNITS

1 milligram per liter.....	=	1 part per million @ 4°C
1 kilogram.....	=	2.205 pounds
1 pound.....	=	453.6 grams
1 grain per gallon.....	=	17.12 parts per million
1 grain per gallon.....	=	142.9 pounds per million gallons
1 part per million.....	=	0.0584 grain per gallon
1 gallon.....	=	231 cubic inches
1 cubic foot.....	=	7.48 gallons
1 cubic foot of water.....	=	62.4 pounds
1 gallon of water.....	=	8.34 pounds
1 gallon.....	=	3.785 liters
1 liter.....	=	0.2642 gallon
1 liter.....	=	1.057 quarts
1 liter.....	=	61.02 cubic inches
1 inch.....	=	2.54 centimeters
1 centimeter.....	=	0.3937 inch
1 cubic foot per second.....	=	646,300 gallons per 24 hours
1 cubic foot per second.....	=	449 gallons per minute
1,000,000 gallons per 24 hours..	=	1.547 cubic feet per second
1,000,000 gallons per 24 hours..	=	694 gallons per minute
1 part per million.....	=	8.34 pounds per million gallons
1 pound per million gallons.....	=	0.1199 parts per million
1 acre.....	=	43,560 square feet
1 gram.....	=	15,432 grains

UNITS (Continued)

1 pound.....	=	7000 grains of wheat
1 meter.....	=	39.37 inches
1 cubic centimeter.....	=	0.0610 cubic inch
1 cubic inch.....	=	16.387 cubic centimeters
1 quart.....	=	0.946 liter
1 gram.....	=	0.0353 ounce
1 ounce.....	=	28.3495 grams

Centigrade temperature = (Fahrenheit - 32) x 5/9

Fahrenheit temperature = (Centigrade x 9/5) + 32

CONVERSION TABLE

G.P.M.	G.P.D.	C.F.S.	M.G.D.
10	14,400	0.022	.014
20	28,800	0.045	.028
30	43,200	0.067	.043
40	57,600	0.089	.057
50	72,000	0.111	.072
75	108,000	0.167	.108
100	144,000	0.223	.144
125	180,000	0.279	.180
150	216,000	0.334	.216
175	252,000	0.390	.252
200	288,000	0.446	.288
250	360,000	0.557	.360
300	432,000	0.668	.432
350	504,000	0.780	.504
400	576,000	0.891	.576
450	648,000	1.00	.648
500	720,000	1.11	.720
550	792,000	1.23	.792
600	864,000	1.34	.864
650	936,000	1.45	.936
700	1,008,000	1.56	1.00
750	1,080,000	1.67	1.08
800	1,152,000	1.78	1.15
850	1,224,000	1.89	1.22
900	1,296,000	2.01	1.29
950	1,368,000	2.12	1.36
1000	1,440,000	2.23	1.44
1200	1,728,000	2.67	1.72
1400	2,016,000	3.12	2.02
1600	2,304,000	3.57	2.30
1800	2,592,000	4.01	2.59
2000	2,880,000	4.46	2.88

G.P.M. - U.S. Gallons per Minute
G.P.D. - U.S. Gallons per 24-hour Day
C.F.S. - Cubic Feet per Second
M.G.D. - Million Gallons per Day

DISCHARGE FROM A PARSHALL FLUME

Gage Reading-Inches	Discharge in cu. ft. per sec. for various throat widths			
	3 Inch	6 Inch	9 Inch	1 Foot
1 3/16	.028	.05	.09	----
1 5/16	.033	.06	.10	----
1 7/16	.037	.07	.12	----
1 9/16	.042	.08	.14	----
1 11/16	.047	.09	.15	----
1 13/16	.053	.10	.17	----
1 15/16	.058	.11	.19	----
2 1/16	.064	.12	.20	----
2 3/16	.070	.14	.22	----
2 1/4	.076	.15	.24	----
2 3/8	.082	.16	.26	.35
2 1/2	.089	.18	.28	.37
2 5/8	.095	.19	.30	.40
2 3/4	.102	.20	.32	.43
2 7/8	.109	.22	.35	.46
3	.117	.23	.37	.49
3 1/8	.124	.25	.39	.51
3 1/4	.131	.26	.41	.54
3 3/8	.138	.28	.44	.58
3 1/2	.146	.29	.46	.61
3 5/8	.154	.31	.49	.64
3 3/4	.162	.32	.51	.68
3 13/16	.170	.34	.54	.71
3 15/16	.179	.36	.56	.74
4 1/16	.187	.38	.59	.77
4 3/16	.196	.39	.62	.80
4 5/16	.205	.41	.64	.84
4 7/16	.213	.42	.67	.88
4 9/16	.222	.45	.70	.92
4 11/16	.231	.47	.73	.95
4 13/16	.241	.48	.76	.99
4 15/16	.250	.50	.78	1.03
5 1/16	.260	.52	.81	1.07
5 3/16	.269	.54	.84	1.11
5 1/4	.279	.56	.87	1.15
5 3/8	.289	.58	.90	1.19
5 1/2	.299	.61	.94	1.23
5 5/8	.309	.63	.97	1.27
5 3/4	.319	.65	1.00	1.31
5 7/8	.329	.67	1.03	1.35

DISCHARGE FROM A PARSHALL FLUME (Continued)

Gage Reading-Inches	Discharge in cu. ft. per sec. for various throat widths			
	3 Inch	6 Inch	9 Inch	1 Foot
6	.339	.69	1.06	1.39
6 1/8	.350	.71	1.10	1.44
6 1/4	.361	.73	1.13	1.48
6 3/8	.371	.76	1.16	1.52
6 1/2	.382	.78	1.20	1.57
6 5/8	.393	.80	1.23	1.62
6 3/4	.404	.82	1.26	1.66
6 13/16	.415	.85	1.30	1.70
6 15/16	.427	.87	1.33	1.75
7 1/16	.438	.89	1.37	1.80
7 3/16	.450	.92	1.40	1.84
7 5/16	.462	.94	1.44	1.88
7 7/16	.474	.97	1.48	1.93
7 9/16	.485	.99	1.51	1.98
7 11/16	.497	1.02	1.55	2.03
7 13/16	.509	1.04	1.59	2.08
7 15/16	.522	1.07	1.63	2.13
8 1/16	.534	1.10	1.66	2.18
8 3/16	.546	1.12	1.70	2.23
8 1/4	.558	1.15	1.74	2.28
8 3/4	.571	1.17	1.78	2.33
8 1/2	.584	1.20	1.82	2.38
8 5/8	.597	1.23	1.86	2.43
8 3/4	.610	1.26	1.90	2.48
8 7/8	.623	1.28	.194	2.53
9	.636	1.31	1.98	2.58
9 1/8	.649	1.34	2.02	2.63
9 1/4	.662	1.36	2.06	2.68
9 3/8	.675	1.39	2.10	2.74
9 1/2	.689	1.42	2.14	2.80
9 5/8	.702	1.45	2.18	2.85
9 3/4	.716	1.48	2.22	2.90
9 13/16	.730	1.50	2.27	2.96
9 15/16	.744	1.53	2.31	3.02
10 1/16	.757	1.56	2.35	3.07
10 3/16	.771	1.59	2.39	3.12
10 5/16	.786	1.62	2.44	3.18
10 7/16	.800	1.65	2.48	3.24
10 9/16	.814	1.68	2.52	3.29
10 11/16	.828	1.71	2.57	3.35
10 13/16	.843	1.74	2.61	3.41
10 15/16	.858	1.77	2.66	3.46
11 1/16	.872	1.81	2.70	3.52
11 3/16	.887	1.84	2.75	3.58
11 1/4	.902	1.87	2.79	3.64
11 3/8	.916	1.90	2.84	3.70

DISCHARGE FROM A PARSHALL FLUME (Continued)

Gage Reading-Inches	Discharge in cu. ft. per sec. for various throat widths			
	3 Inch	6 Inch	9 Inch	1 Foot
11 1/2	.931	1.93	2.88	3.76
11 5/8	.946	1.97	2.93	3.82
11 3/4	.961	2.00	2.98	3.88
11 7/8	.977	2.03	3.02	3.94
12	.992	2.06	3.07	4.00

DISCHARGE FROM TRIANGULAR NOTCH WEIRS WITH END CONTRACTIONS

Head In Inches	Flow In Gallons Per Minute 90° Notch	60° Notch
1	2.19	1.27
1 1/4	3.83	2.21
1 1/2	6.05	3.49
1 3/4	8.89	5.13
2	12.4	7.16
2 1/4	16.7	9.62
2 1/2	21.7	12.5
2 3/4	27.5	15.9
3	34.2	19.7
3 1/4	41.8	24.1
3 1/2	50.3	29.0
3 3/4	59.7	34.5
4	70.2	40.5
4 1/4	81.7	47.2
4 1/2	94.2	54.4
4 3/4	108	62.3
5	123	70.8
5 1/4	139	80.0
5 1/2	156	89.9
5 3/4	174	100
6	193	112
6 1/4	214	124
6 1/2	236	136
6 3/4	260	150
7	284	164
7 1/4	310	179
7 1/2	338	195
7 3/4	367	212
8	397	229
8 1/4	429	248
8 1/2	462	267
8 3/4	498	287
9	533	308
9 1/4	571	330
9 1/2	610	352
9 3/4	651	376
10	694	401
10 1/2	784	452
11	880	508
11 1/2	984	568
12	1094	632

REPORT OF LABORATORY RESULTS

Prepared June 22, 1970

PARAMETER	REPORT TO NEAREST:
Dissolved Oxygen	0.1 mg/l
pH	0.1 units
Conductance	
0-999 umhos	1 umhos
>1000 umhos	5 umhos
Turbidity	
0.0-1.0	0.05 Jackson units
1-10	0.1 Jackson units
10-40	1 Jackson units
40-100	5 Jackson units
100-400	10 Jackson units
400-1000	50 Jackson units
>1000	100 Jackson units
Alkalinity	1 mg/l
Hardness	1 mg/l
Chloride	
0.0-1.0	0.1 mg/l
>1	1 mg/l
Sulfate	
0-9.9	0.1 mg/l
10-1000	1 mg/l
>1000	10 mg/l
Phosphorus (Total and Dissolved)	
0-9.99	0.01 mg/l
>10.0	0.1 mg/l
Ortho	
0-9.99	0.01 mg/l
>10.0	0.1 mg/l
Nitrogen	
Total Kjeldahl	
0-2	0.01 mg/l
2-10	0.1 mg/l
>10	1 mg/l

REPORTING OF LABORATORY RESULTS (Continued)

PARAMETER	REPORT TO NEAREST:
Ammonia	
0-2	0.01 mg/l
2-10	0.1 mg/l
>10	1 mg/l
Nitrogen	
Nitrates	
0-10.0	0.01 mg/l
>10.0	0.1 mg/l
Nitrites	
0-10.0	0.01 mg/l
>10.0	0.1 mg/l
Solids	
1-1000	1 mg/l
>1000	10 mg/l
Biochemical Oxygen Demand	
0-9.9	0.1 mg/l
10-499	1 mg/l
500-1000	10 mg/l
>1000	100 mg/l
Chemical Oxygen Demand	
0-9.9	0.1 mg/l
10-1000	1 mg/l
>1000	10 mg/l
Sodium and Potassium	
0-9.9	0.1 mg/l
10-1000	1 mg/l
Fluoride	0.1 mg/l
Phenols (Chloroform Extraction and 50 mm Cell)	
0-99	1 mg/l
100-1000	10 mg/l
>1000	100 mg/l
Cyanide	
0-9.99	0.01 mg/l
10.0-99.9	0.1 mg/l

REPORTING OF LABORATORY RESULTS (Continued)

PARAMETER	REPORT TO NEAREST:
Organic Carbon	
0-9	0.1 mg/l
10-100	1 mg/l
>100	5 mg/l
Oil and Grease	
0-99	1 mg/l
100-1000	10 mg/l
>1000	100 mg/l

SAMPLE COLLECTION AND PRESERVATION

ANALYSES	CONTAINER	PRESERVATION	MAXIMUM HOLDING PERIOD	COLOR IDENTIFICATION (TAG)
Nutrients Phosphorus Nitrogen	1-Cubitainer	40 mg/l $HgCl_2$ (4 mls of 1% Sol) at 4°C	7 days	Yellow
Cyanide	1-Cubitainer	NaOH to pH 10 or grtr.	24 hours	Green
Phenolics	1-Cubitainer	1 gram of $CuSO_4$+H_2PO_4 to pH of 4 → 4°C or 5 ml of 20% $CuSO_4$ 2-3 ml of 80% H_3PO_4	24 hours	Red
Acidity or Alkalinity	1-Cubitainer	4°C	24 hours	Manila (2 sides)
Calcium		None Required	--------	
Chloride		"	--------	
Fluoride		"	--------	
Hardness		"	--------	
pH		None Available	--------	
Solids		"	--------	
Sp Cond		"	--------	
Sulfates		4°C	7 days	
Turbidity		"	--------	
Bact. Sample	Bact. Bottle	4°C	6 hours	Manila (1 side)
Metals (Total)	1-Cubitainer 4 oz. bottle	5 ml conc. HNO_3 0.6 ml conc. HNO_3	6 months	White
BOD_2 & 5	1 gallon jug	4°C	8 hours	Manila (1 side)
DO	DO Bottle	Flocculated with Hach Reagents 4°C- dark	6 hours	Manila (1 side)
COD Organic Carbon	1-Cubitainer 4 oz. bottle	2 ml conc HCl 0.25 ml conc HCl	7 days	Pink
Oil and Grease	1 L Glass stoppered bottle	2 ml H_2SO_4/liter	24 hours	Manila (1 side)
Biology Samples	1-Cubitainer	35 ml of 4% Formalin	Forever	Blue

GLOSSARY

1. Anhydrous - means dry or free from water.

2. Buffer - is a chemical substance which is used to prevent or reduce changes in the pH.

3. Burette - (volume burette) is a graduated apparatus used to measure accurately the volume of a solution delivered.

4. Caustic - highly alkaline like lye.

5. Desiccator - is a container in which heated objects which are to be weighed are allowed to cool down to room temperature. It should contain a chemical (such as calcium chloride or silicagel) which will pick up water from the air which entered the desiccator.

6. Fixed Solids - (total or suspended) are the residue after ignition. This may also be called ash. It is inorganic in nature.

7. gpg - grains per gallon.

8. Ignite - means to burn. In this case it means burning off the organic material leaving a white ash.

9. Mix thoroughly - if a volumetric flask is used, stopper and invert 15 times. Otherwise, mix with a clean glass rod at least 5 minutes.

10. ml - is short for milliliter. cc is short for cubic centimeter. They are both units of volume. 1.00000 ml = 1.00003cc. For practical purposes, ml and cc are the same. There are 1000 mls in a liter.

11. mg - (or milligram) is $1/1000^{th}$ of a gram. To change grams to mg multiply by 1000.

12. mg/l - is milligrams per liter, a weight to volume ratio. Strictly, mg/l is not equal to ppm, but for practical purposes they are the same. Standard Methods recommends using mg/l.

13. ppm - is short for parts per million. This is a weight to weight ratio meaning one part of one substance in one million parts of the total.

14. Reagent - is a substance used to act upon another substance in a chemical reaction. In this case, the reagents are solutions which carry active ingredients of a definite strength.

GLOSSARY (Continued)

15. Saturated - in such a condition (whether in solid, gaseous, or liquid state) that another material held within a given state is in an amount such that no more of such material can be held within in the same state.

16. Septicity - is a condition of decomposition in which there is no DO and odors may be produced.

17. Solvent - liquid used to dissolve a substance.

18. Stability - the ability of any substance, such as wastewater, chemicals, or digested sludge, to resist change though it may change slightly at different times of the year.

19. Thio - is short for sodium thiosulfate solution.

20. Titration - is the operation of accurately adding a solution of known concentration (standard solution) to a solution which is being tested. The exact amount to add is determined with the aid of another substance (indicator) which will change color at the proper time. This point of change is called the "end-point."

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We have modified the procedures and equipment discussed in the Missouri manual in order to keep up with the rapidly changing and complex pollution problems, and tied together our experiences in the water pollution field and made improvements with up-to-date methods and instrumentation. This should further reduce the intricate parts of the tests and upgrade the accuracy and precision of the analysis. Without using the Missouri Water Pollution Manual as a format, this would not have been possible.